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Characterizaion of Nine High-Mileage Catalytic Converters for EPA

Automotive Testing Labs.
Aurora, CO

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Automotive Testing Laboratories, Inc.

CHARACTERIZATION OF NINE HIGH-MILEAGE CATALYTIC CONVERTERS FOR EPA

(Contracted through Automotive Testing Laboratories P. O. No. 2628)

R. G. Lyben and L. M. Niebylski

September 1979

Ethel Corporation Research Laboratories
Ferndale, Michigan 48220

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This study was a pilot project designed to analyze the efficiency and condition of in-use catalysts on high-mileage vehicles. These 1975 model year vehicles were originally all public-owned and were obtained from an earlier EPA project (Restorative Maintenance Evaluation of High-Mileage, Catalyst-Equipped Vehicles, conducted for EPA by Automotive Testing Laboratories, Inc., EPA Contract #68-03-2413). Tables I and II present descriptions, miles accumulated and FTP results for these nine vehicles from this past report.

For the current project the catalysts were removed from the vehicles and tested for conversion efficiency and pressure drop. The catalysts were then subjected to visual, chemical and physical analysis to provide data on their in-use condition.

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FOREWARD

This study was a pilot project designed to analyze the efficiency and condition of in-use catalysts on high-mileage vehicles. These 1975 model year vehicles were originally all public-owned and were obtained from an earlier EPA project (Restorative Maintenance Evaluation of High-Mileage, Catalyst - Equipped Vehicles, conducted for EPA by Automotive Testing Laboratories, Inc., EPA Contract #68-03-2413). Tables I and II present descriptions, miles accumulated and FTP results for these nine vehicles from this past report.

For the current project the catalysts were removed from the vehicles and tested for conversion efficiency and pressure drop. The catalysts were then subjected to visual, chemical and physical analysis to provide data on their in-use condition.

Table I presents descriptions and miles accumulated for the original nine vehicles from the earlier report.

TABLE I

High Mileage Vehicles for Catalyst Evaluation

Veh. #	Make	Model	Miles Accumulated	CID	IW	Trans.	Engine Family
511	Plymouth	Duster	138,831	225	3500	A	F-RG-CII
512	Dodge	Charger	75,064	360	4500	A	F-LA2L-C
513	Plymouth	St.Wagon	71,026	318	5000	A	F-LA2-6C
524	Mercury	Monarch	103,977	250	5000	M3	2501CEF
525	Ford	Custom	108,238	400	5000	A	351M/400"E"
526	Ford	LTD	111,512	351	4000	A	351M/4001CET
537	Chev	Nova	89,691	260	3500	A	10F13
538	Chev	Malibu	107,979	350	4500	A	10J23
539	Buick	St.Wagon	137,751	350	4500	A	40J43

Table II presents the composite FTP emission and fuel economy results for the high-mileage vehicles from the earlier report. Testing was performed after emission control repair, removal of catalyst and catalyst replacement.

Table II
FTP Composite Results for High-Mileage Vehicles

Test Conditions:

(1) measured with high mileage catalyst after emission component repair and vehicle maintenance.
(2) measured with catalyst removed
(3) measured after the original catalyst was replaced with a new catalyst

Veh#	Test Condition	g/mi HC	g/mi CO	g/mi NOx	MPGu
511	(1)	3.00	22.9	2.54	17.11
	(2)	3.15	29.8	2.10	18.24
	(3)	1.65	20.1	1.34	17.49
512	(1)	3.75	16.6	2.08	11.05
	(2)	8.97	16.8	1.79	12.01
	(3)	.93	21.8	.67	11.17
513	(1)	.74	4.6	2.12	11.46
	(2)	1.68	28.5	1.78	11.67
	(3)	.62	14.3	1.34	11.69
524	(1)	1.52	8.0	2.68	17.79
	(2)	2.09	20.7	2.41	18.17
	(3)	.6	4.8	2.81	17.73
525	(1)	3.07	35.2	3.06	6.61
	(2)	2.90	17.3	4.00	10.86
	(3)	2.13	14.3	4.44	9.85
526	(1)	2.04	11.0	6.38	11.63
	(2)	2.84	10.3	6.76	11.73
	(3)	2.05	8.0	6.21	11.64
537	(1)	1.14	7.5	1.17	17.67
	(2)	3.35	9.1	1.25	18.56
	(3)	.64	5.6	1.08	18.41
538	(1)	1.90	12.9	1.52	13.05
	(2)	4.07	21.0	1.42	12.66
	(3)	.69	7.3	1.37	12.36
539	(1)	1.41	11.9	1.87	12.53
	(2)	2.31	21.6	1.86	11.89
	(3)	.59	8.4	1.49	11.88

CHARACTERIZATION OF NINE CATALYTIC CONVERTERS REMOVED FROM 1975 PASSENGER CARS

INTRODUCTION

This report is divided into two sections authored by two different people. The first section deals with dynamometer tests made to obtain relative emission data, photographs of cut-apart converter cases, semi-quantitative analysis by emission spectroscopy of major elements present on the inlet faces of the converters and quantitative lead analysis of one converter which appeared to have been run on leaded fuel. The second section deals with data obtained using X-ray diffraction, B. E. T. measurements of surface area, differential thermal analysis, energy dispersive analysis and scanning electron microscope analysis.

SECTION I

Report by R. G. Lyben

A. Description of Dynamometer Tests

A 400-CID Ford V-8 engine equipped with an adjustable carburetor and coupled to a dynamometer is used to supply a controlled exhaust gas mixture to the converter under test. Table 1 gives the operating parameters of the engine. Figure 1 is a schematic drawing of the test piping. Photos 1 and 2 show a converter attached to the engine for testing. Photo 3 shows the emission measuring consoles used in this work.

The test procedure used is detailed below:

1. Warm up the engine with the converter installed but bypassed. The two valves attached to the converter are closed and the valve in the bypass line is open while the engine is brought up to equilibrium temperature at 50 mph road-load. The air/fuel ratio is adjusted to 16.0 at which level 1.9 - 2.0% oxygen is present in the exhaust gas.
2. When conditions have stabilized, the valves to the converter are opened and the bypass valve is closed. Temperatures, carbon-monoxide and hydrocarbons are continuously recorded for samples drawn from the converter inlet and outlet taps. These measurements are continued until readings show no further change.
3. Pressure drop across the converter is measured at 50 mph road-load and then the engine is adjusted to 3800 rpm WOT where another pressure drop measurement is made. A 30" mercury manometer is used to make these pressure measurements.
4. The recorded charts are then examined to determine the time and temperature it takes to reach 50% hydrocarbon conversion. These data can be related to warm-up time. With some of the converters,

the 50% point is never reached. With 2% oxygen in the exhaust, CO levels are too low to be significant and very little reduction in CO occurs.

B. Converter Efficiency

Nine converters removed from high-mileage 1975 passenger cars were tested. Three converters were double-biscuit monoliths removed from Chrysler vehicles. Three single-biscuit monolithic converters were from Ford vehicles. The Ford converters of this model year were only installed in one leg of the exhaust system, thus only receiving one-half of the exhaust gas. When analyzing the Ford data, this should be kept in mind. The last three converters came from GM cars. These were all 260 cubic inch containers. Two were filled with spherical catalyst and one was filled with cylindrical catalyst pellets.

1. Chrysler Converters - The Chrysler converters have two ceramic biscuits mounted in a single canister. Emission data are given in Table 2. Converters 511 and 513 gave HC reductions of 67 and 68% at equilibrium and appear to be undamaged. Converter 512 had a melted inlet face in the second section. This can be clearly seen in Photo 10. Photos 3-15 show all inlet and outlet faces of the Chrysler converters.

   a. Warm-up Time - This is defined as the time required to reach 50% HC conversion. Catalyst 512 had a maximum conversion of 24% due to the melt.

Cat. #	Time to reach 50% HC Reduction, secs.	Temperature °C Inlet	Temperature °C Outlet
511	62	520	480
512	N. R.	---	---
513	50	515	400

   b. Pressure Drop - As expected, catalyst 512 had the highest pressure drop:

Cat. #	Back Pressure " Hg 50 mph Road Load	 3800 rpm W. O. T.
511	1.6	8.0
512	2.4	11.0
513	1.2	6.2

2. Ford Converters - These are single-section monoliths used only in one leg of the exhaust systems in the 1975 model year. As shown in Figure 2, HC conversions were 55, 17 and 43%. Catalyst 525 was cut in half to exhibit a peculiar melt which began 1/2" from the inlet. Usually, several inches of catalyst are required to build temperatures up to the melting point of the ceramic (see Photo 19). Other photos of Ford inlet and outlet converter faces are shown in Phtos 16-22.
   a. Warm-up Time - Only converter 524 was active enough to reduce hydrocarbons by 50%. This occurred in 55 seconds at an inlet temperature of 515°C and 410°C outlet.
   b. Pressure Drop - Pressure drops for the Ford catalysts are given below:

Cat. #	Back Pressure " Hg 50 mph Road Load	 3800 rpm W. O. T.
524	0.8	5.1
525	2.4	13.0
526	0.6	4.6

3. General Motors Converters - These 260-in.^{3} converters utilize alumina spheres or pellets. Bacause of the greater mass involved, one would expect longer warm-up times before conversion starts. The GM catalysts gave conversions of 86, 57 and 74%. Converter 538 had the lowest conversion efficiency, longest 50% HC conversion time and largest pressure drop of the three. Inspection of the outlet screen showed partial plugging due to attrition of the catalyst as shown in Photos 23-26.

a. Warm-up Time - The times to reach 50% HC reduction are given below:

Cat. #	Time to reach 50% HC Reduction, secs.	Temperature °C Inlet	Temperature °C Outlet
537	<30	482	75
538	150	545	440
539	75	530	240

Catalyst 537 was exceptionally active.

b. Back Pressure - Catalyst 538 had the highest back pressure due to partial screen blockage.

Cat. #	Back Pressure "Hg 50 mph Road Load	Back Pressure "Hg 3800 rpm W. O. T.	
537	0.8	5.8	cylinders
538	2.4	13.8	spheres
539	1.0	6.8	spheres

C. Elemental Analysis of Converter Inlet Faces

Order of magnitude analyses were made of samples removed from the inlet faces of the monolithic converters. Aliquot samples were also taken from the GM pellet-filled converters. Monolith samples were obtained by drilling 3/8" holes to a depth of 1/2". All samples were ground to a fine powder and analyzed using Emission Spectroscopy.

The source of the elements found would be mainly from the fuel and oil. For example, unleaded fuels still contain about 0.02 gram lead/gallon on average. Thus in 100,000 miles of driving on unleaded fuel, assuming 15 miles per gallon, 133 grams of lead would pass through the engine. Since the alumina and silica catalyst supports are good adsorbents of lead halides in the vapor state, it is not surprising to find appreciable amounts of lead in all samples.

1. Chrysler Converters - Table 3 lists the elements found on the inlet converter faces. Since the Chrysler converters are double-section monoliths, two analyses are shown for each converter. Catalyst 512 would appear to have been exposed to leaded fuel. Additional analyses for lead were made on converters 512 and 513 by Atomic Absorption. Percent lead found was:

	Converter 512	Converter 513
Inlet #1	29.10	2.21
Inlet #2	3.41	0.58

2. Ford Converters - The Ford converters are single-section monoliths and were used in only one leg of the exhaust system in the 1975 model year. Converter 525 suffered a peculiar melt that started about 1/2" from the inlet surface. The lead content of all three inlet faces is in the 1-10% range so it is unlikely that any of these ran on leaded fuel. Larger amounts of calcium, copper, titanium and zinc were found on the melted converter than on the other two. The elemental analyses are given in Table 4.
3. GM Converters - One of the converters was filled with cylindrical pellets while the other two contained catalyst spheres. Table 5 gives elemental compositions for the three converters. The support material appears to be of different composition for the cylinders. The cylinder-filled converter (#537) also was superior in conversion of HC, warm-up time and back pressure.

D. Conclusions

It would be difficult to reach firm conclusions on the basis of testing only nine converters. We can, however, compare the nine converters tested.

1. The GM converters as a group were better than the Chrysler converters, which in turn were better than the Ford converters.

2. One each of the Chrysler and Ford converters had ceramic melts. This occurs when ignition malfunctions and unburned gasoline is pumped into the exhaust system. It takes only a few minutes to reach ceramic melting point temperatures under this condition.
3. One GM converter had high back-pressure caused by small catalyst particles lodging in the output screen due to attrition of the catalyst.
4. One of the Chrysler cars appears to have been driven on leaded gasoline since the lead found on the face was more than 10 times greater than on any of the other monoliths.

TABLE 1

1973 Ford 400-CID V-8
Operating Parameters

Carburetor: D3MF-EA (Ford)
ASTM manual STP 315F
Sequence V-C - carburetor modification with V50F main jets and power valve drilled to 0.078"

Distributor Timing:
6° BTC @ 625 rpm
26° BTC @ 1900 rpm

50-mph Road Load:
1900 ± 25 rpm @ 37# Beam = 70.3 BHP

A/F	15	16	17	18
HC, ppm	146.0	105.0	81.0	-----
CO %	0.309	0.180	0.089	0.003
CO_2 %	14.60	13.59	12.83	12.20
O_2 %	0.7	1.9	3.15	4.12
NO_x, ppm	2870	2917	1945	-----

TABLE 2

Equilibrium Conversion Data (50 mph Road Load)

Converters Removed from High-Mileage 1975 Cars

Cat. #	°C In	°C Out	HC ppm In	HC ppm Out	% Reduct.	CO % In	CO % Out	O_2 %	A/F	
Chrysler Monoliths:										
511	552	540	78.58	26.19	66.7	0.067	0.056	1.95	15.92	
512	565	526	68.28	52.21	23.5	0.081	0.064	2.08	16.06	
513	563	551	75.81	23.94	68.4	0.075	0.063	2.00	15.91	
Ford Monoliths:										
524	553	535	77.80	35.10	54.9	0.067	0.063	1.95	15.97	
525	552	515	80.48	66.74	17.1	0.064	0.064	1.95	15.90	
526	550	520	81.65	46.66	42.9	0.070	0.060	1.95	15.90	
GM Pellets:										
537	562	537	87.00	12.00	86.2	0.064	0.053	1.93	15.90	cylinders
538	558	516	86.35	36.95	57.2	0.056	0.053	1.98	15.95	spheres
539	555	520	90.86	23.93	73.7	0.075	0.053	1.95	15.95	spheres

Vehicle Identification:

#511	Plymouth Duster
#512	Dodge Charger
#513	Plymouth Wagon
#524	Mercury 250-6
#525	Ford 400-CID Custom
#526	Ford LTD
#537	Chevrolet Nova 250-6
#538	Chevrolet Malibu
#539	Buick Century Wagon

RGL:ah

TABLE 3

Elements Found on Inlet Faces of Chrysler Converters

Values are "Order of Magnitude" Percentages

Element	Converter 511		Converter 512		Converter 513	
	Inlet 1	Inlet 2	Inlet 1	Inlet 2	Inlet 1	Inlet 2
Aluminum	>10	>10	>10	>10	>10	>10
Barium	0.1-1	0.1-1	0.01-0.1	0.01-0.1	0.1-1	0.1-1
Berylium	---	---	------	------	---	---
Boron	---	---	------	------	---	---
Calcium	0.1-1	0.1-1	0.1-1	0.1-1	0.1-1	0.1-1
Chromium	0.1-1	0.01-1	0.01-0.1	0.01-1	0.01-0.1	0.01-0.1
Copper	0.01-0.1	0.01-0.1	0.01-0.1	0.001-.01	0.1-1	0.01-0.1
Iron	1-10	1-10	1-10	1-10	1-10	1-10
Lead	1-10	1-10	>10	1-10	1-10	1-10
Magnesium	>10	>10	>10	>10	>10	>10
Manganese	0.1-1	0.1-1	0.1-1	0.1-1	0.1-1	0.1-1
Molybdenum	0.001-.01	0.001-.01	0.001-.01	0.001-.01	0.001-.01	0.001-.01
Nickel	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1
Palladium	----	----	0.01-0.1	0.01-0.1	----	----
Phosphorus	1-10	1-10	1-10	0.1-1	1-10	0.1-1
Platinum	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1
Silicon	>10	>10	>10	>10	>10	>10
Strontium	0.001-.01	0.001-.01	0.001-.01	0.001-.01	0.001-.01	0.001-.01
Tin	0.001-.01	0.001-.01	0.001-.01	0.001-.01	-----	-----
Titanium	0.1-1	0.1-1	0.1-1	0.1-1	0.1-1	0.1-1
Vanadium	0.001-.01	0.001-.01	0.001-.01	0.001-.01	0.001-.01	0.001-.01
Zinc	1-10	0.1-1	1-10	0.01-0.1	0.1-1	0.01-0.1
Lead by A.A.	-----	-----	29.10	3.41	2.21	0.58
HC Reduction	67		24		68	
Warm-up Time, secs.	62		----		50	
Back Pressure WOT	8.0		11.0		6.2	

TABLE 4

Elements Found on Inlet Faces of Ford Converters

Values are "Order of Magnitude" Percentages

Element	Converter 524 Inlet Face	Converter 525 Inlet Face	Converter 526 Inlet Face
Aluminum	>10	>10	>10
Barium	----	0.01.0.1	0.01-0.1
Berylium	----	-----	-----
Boron	----	-----	0.01-0.1
Calcium	0.01-0.1	1-10	0.1-1
Chromium	0.01-0.1	0.1-1	0.01-0.1
Copper	0.01-0.1	1-10	0.1-1
Iron	1-10	>10	1-10
Lead	1-10	1-10	1-10
Magnesium	>10	>10	>10
Manganese	1-10	1-10	1-10
Molybdenum	0.001-.01	0.01-0.1	0.001-.01
Nickel	0.01-0.1	0.1-1	0.001-0.1
Palladium	0.1-1	0.1-1	0.1-1
Phosphorus	1-10	1-10	1-10
Platinum	0.01-0.1	0.01-0.1	0.01-0.1
Silicon	>10	>10	>10
Strontium	0.001-.01	0.001-.01	0.01-0.1
Tin	----	0.01-0.1	----
Titanium	0.1-1	1-10	1-10
Vanadium	0.01-0.1	0.01-0.1	0.01-0.1
Zinc	0.1-1	1-10	0.1-1
HC Reduction	55	17	43
Warm-up Time, secs.	55	--	--
Back Pressure WOT	5.1	13.0	4.6

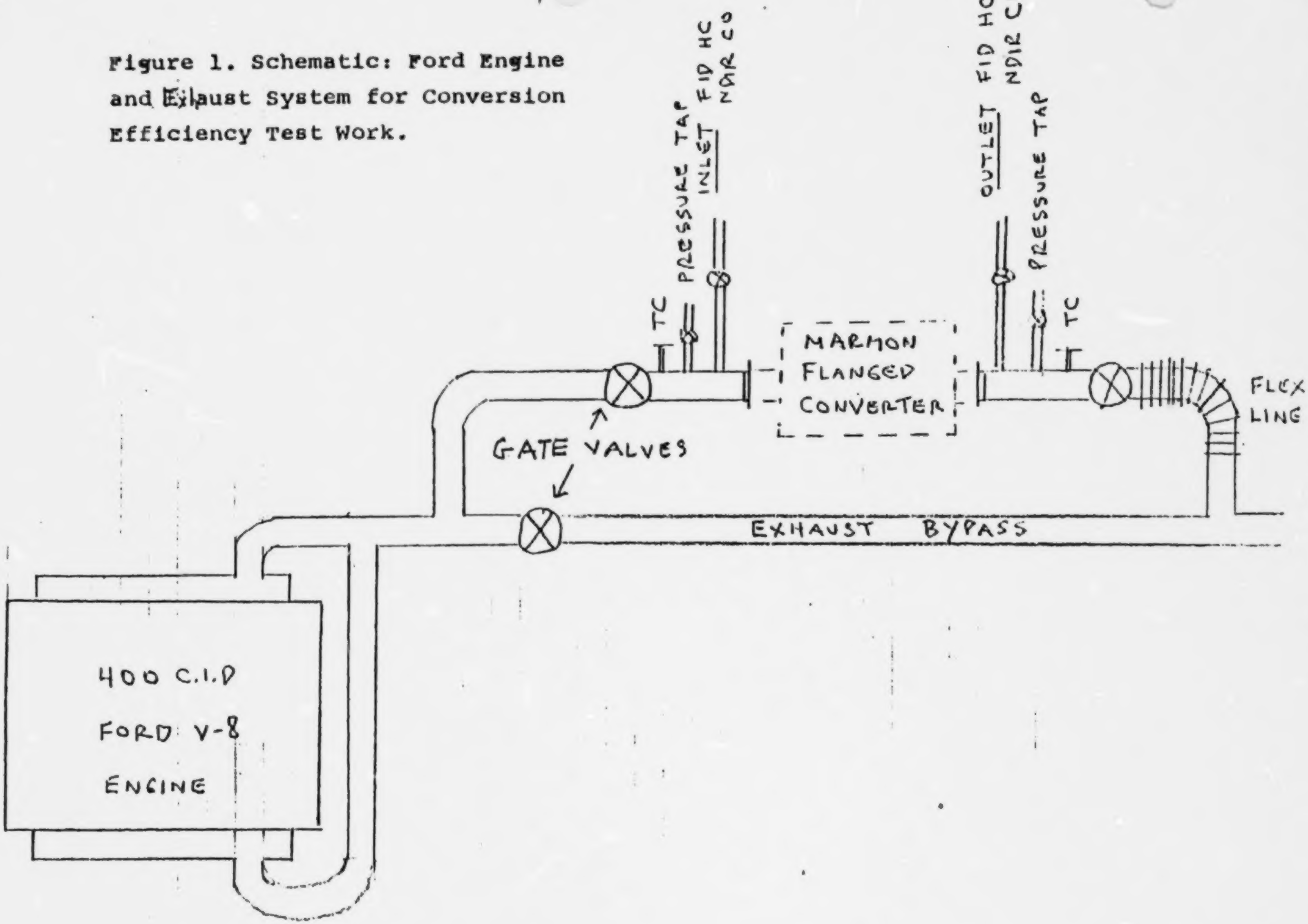
TABLE 5

Elements Found on Aliquot Samples of Catalyst Pellets from GM Converters

Values are "Order of Magnitude" Percentages

Element	Converter 537 Cylinders	Converter 538 Spheres	Converter 538 Spheres
Aluminum	>10	>10	>10
Barium	----	----	----
Berylium	----	0.001-.01	0.001-.01
Boron	----	0.01-0.1	0.01-0.1
Calcium	0.01-0.1	0.01-0.1	0.01-0.1
Chromium	0.01-0.1	0.01-0.1	0.01-0.1
Copper	0.001-.01	0.001-.01	0.001-.01
Iron	0.1-1	0.1-1	0.1-1
Lead	0.1-1	0.1-1	0.1-1
Magnesium	0.01-0.1	0.1-1	0.01-0.1
Manganese	0.1-1	0.1-1	0.1-1
Molybdenum	0.001-.01	0.001-.01	0.01-0.1
Nickel	-----	-----	-----
Palladium	0.1-1	0.1-1	0.1-1
Phosphorus	-----	0.1-1	0.1-1
Platinum	0.01-0.1	0.01-0.1	0.01-0.1
Silicon	0.1-1	0.01-0.1	0.01-0.1
Strontium	-----	-----	-----
Tin	-----	-----	-----
Titanium	0.01-0.1	0.01-0.1	0.01-0.1
Vanadium	-----	0.001-.01	0.001-.01
Zinc	-----	0.1-1	0.1-1
HC Reduction	86	57	74
Warm-up Time, secs.	<30	150	75
Back Pressure WOT	5.8	13.8	6.8

Figure 1. Schematic: Ford Engine and Exhaust System for Conversion Efficiency Test Work.



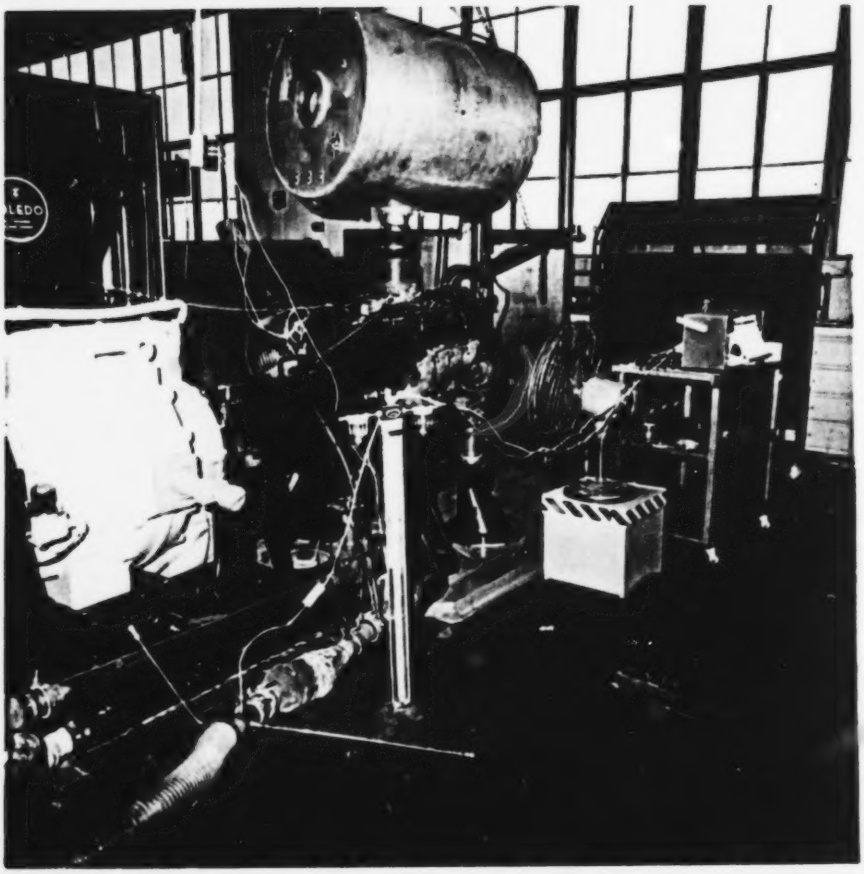


Photo 1. Engine Setup

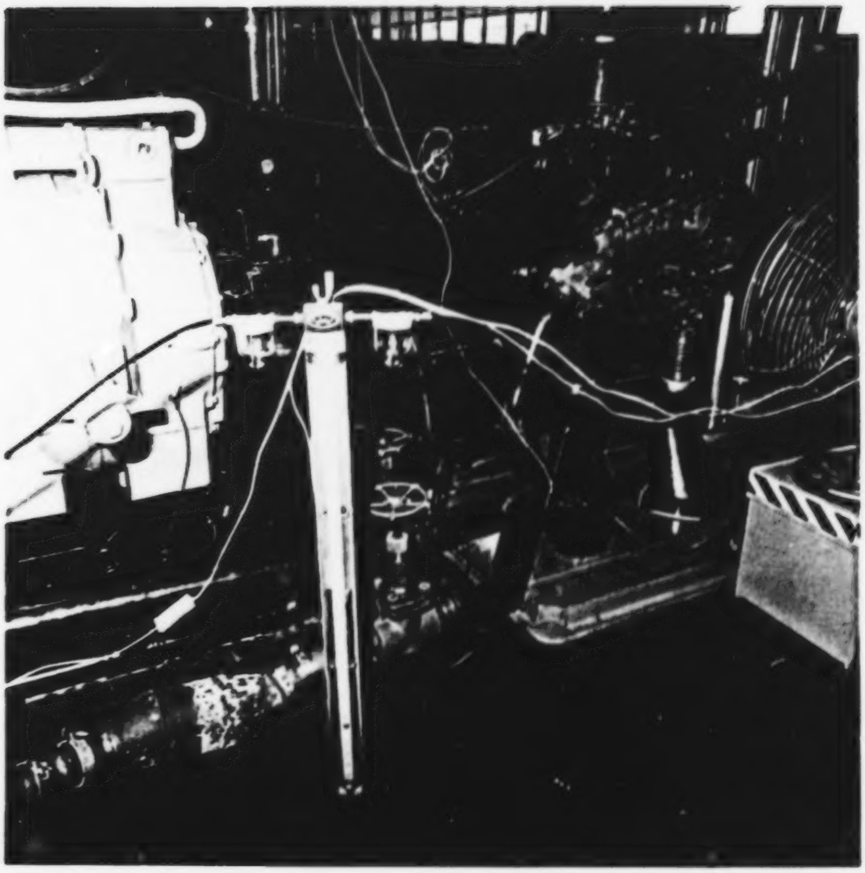


Photo 2. Engine Setup
(Close-up View)

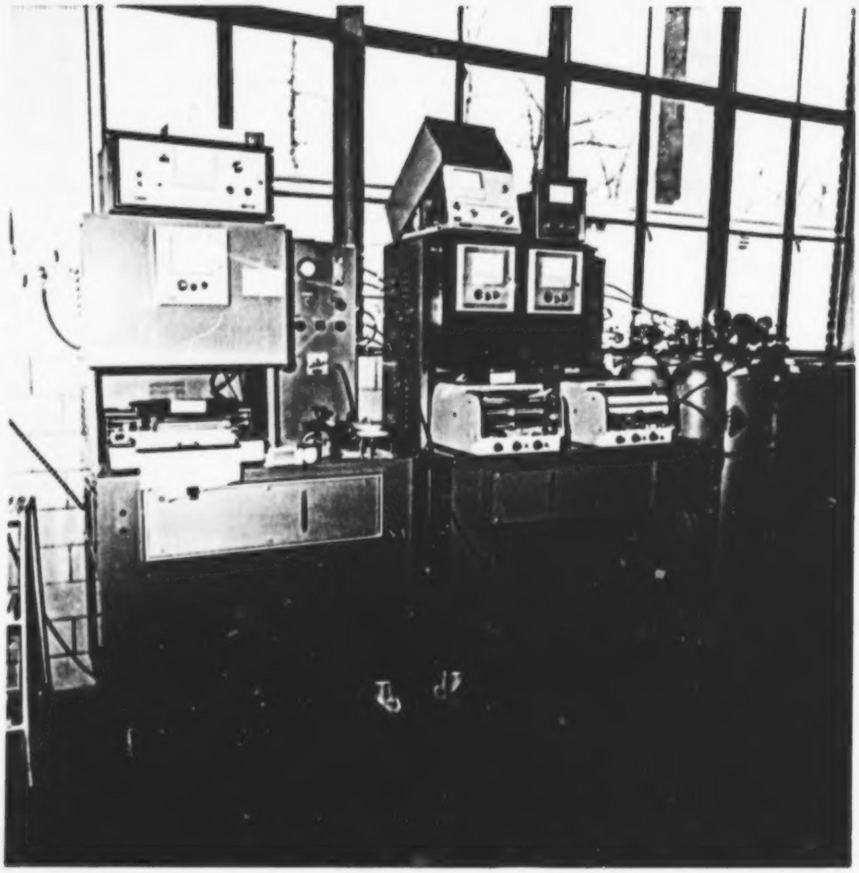


Photo 3. Emission Measuring Consoles

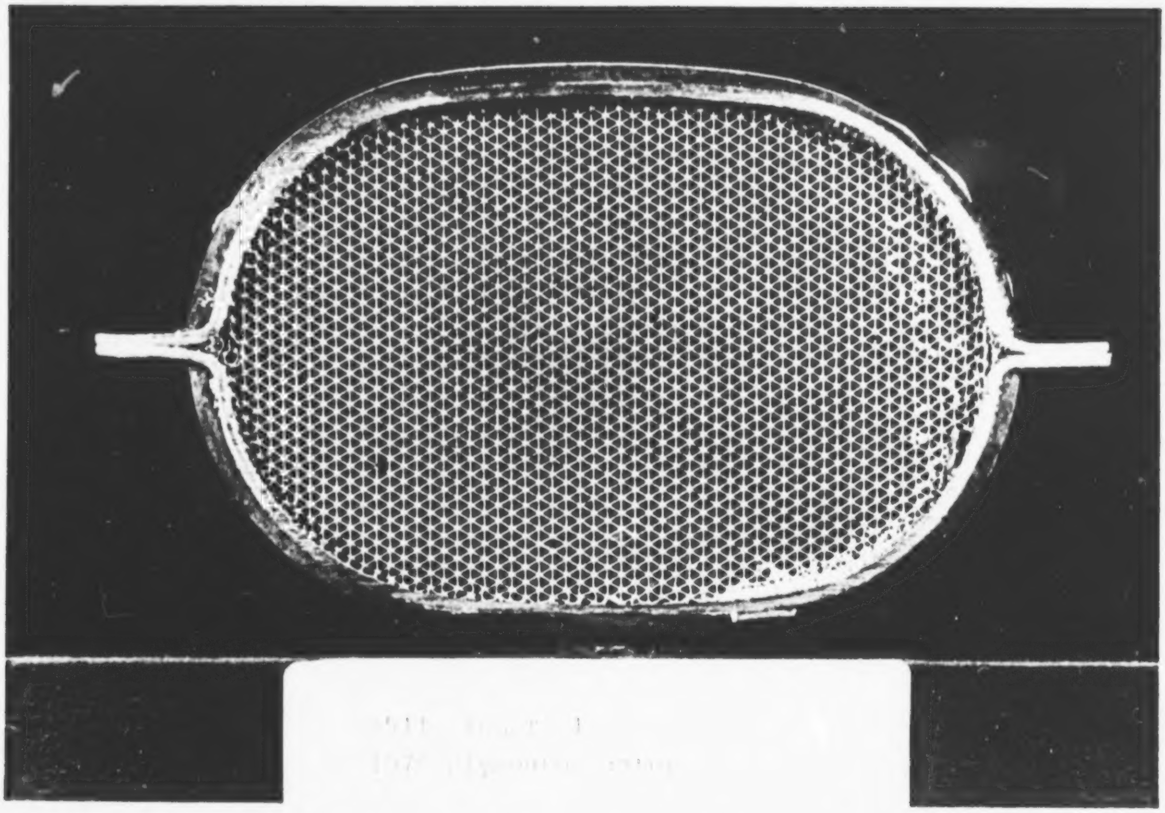


Photo 4. Converter #511 Inlet 1

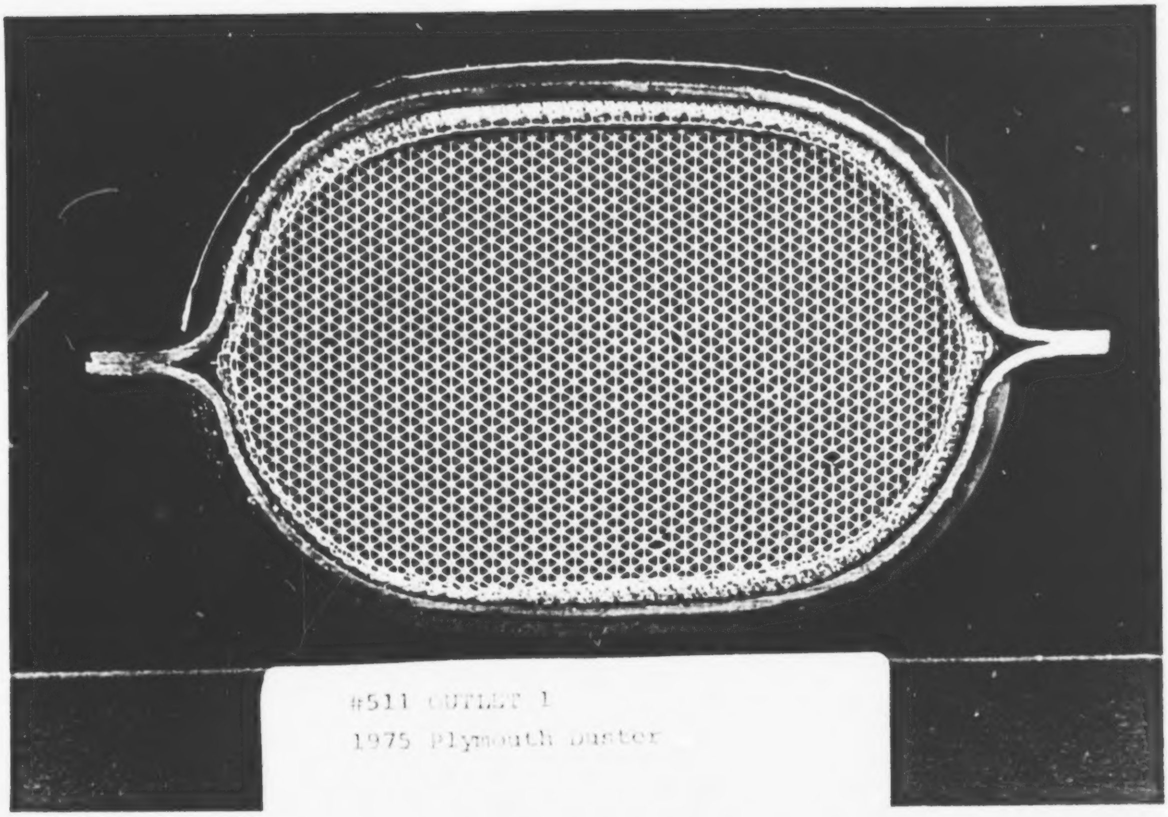


Photo 5. Converter #511 Outlet 1

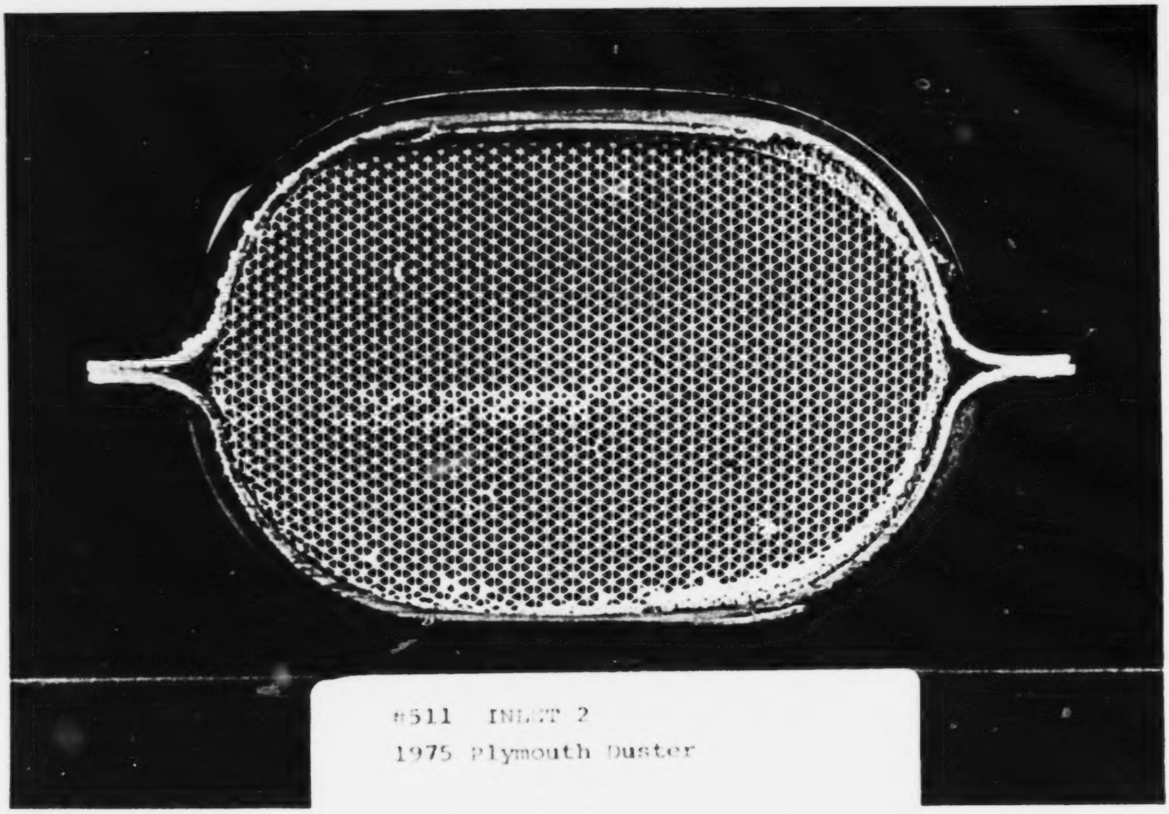


Photo 6. Converter #511 Inlet 2

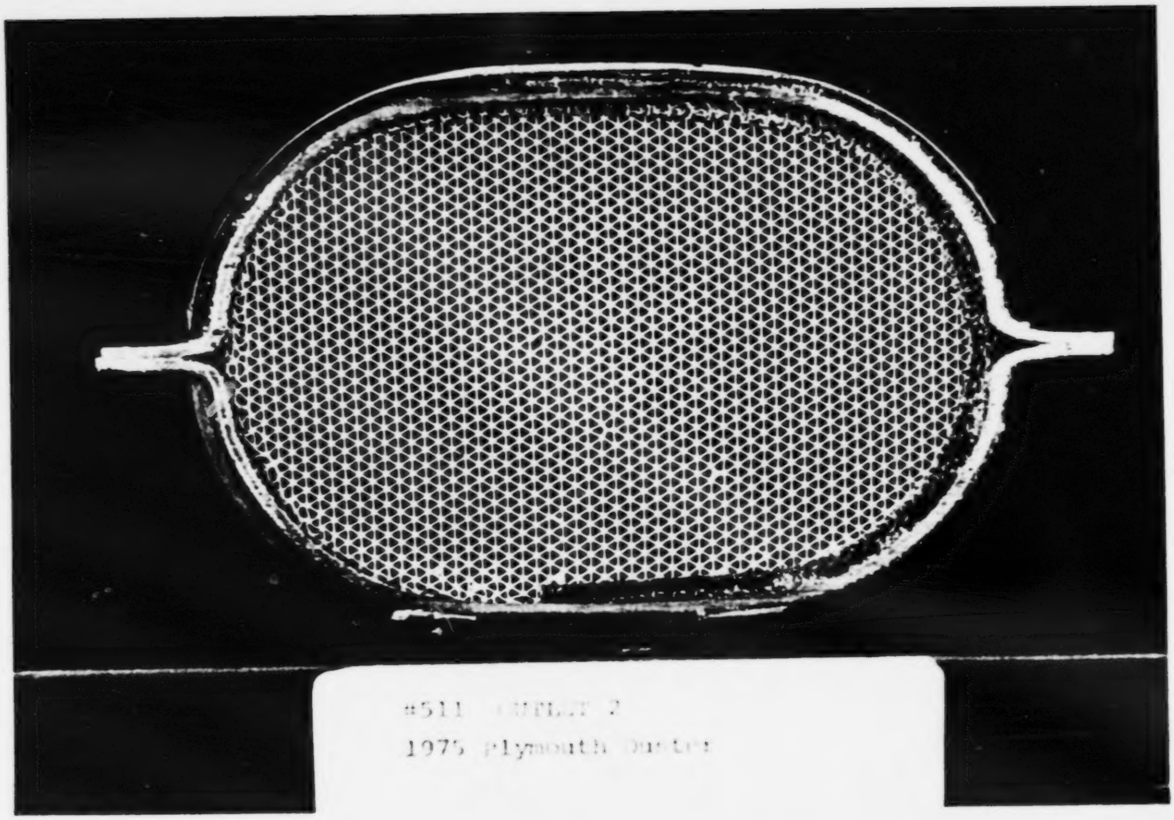


Photo 7. Converter #511 Outlet 2

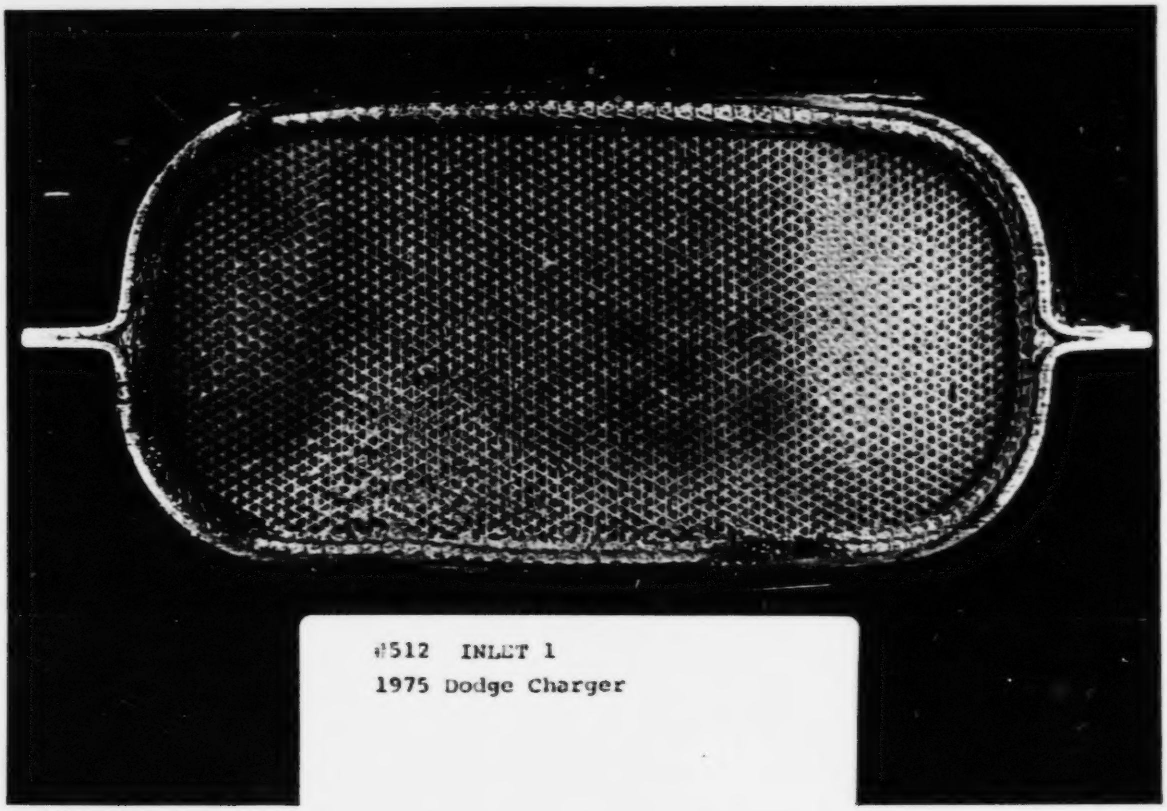


Photo 8. Converter #512 Inlet 1

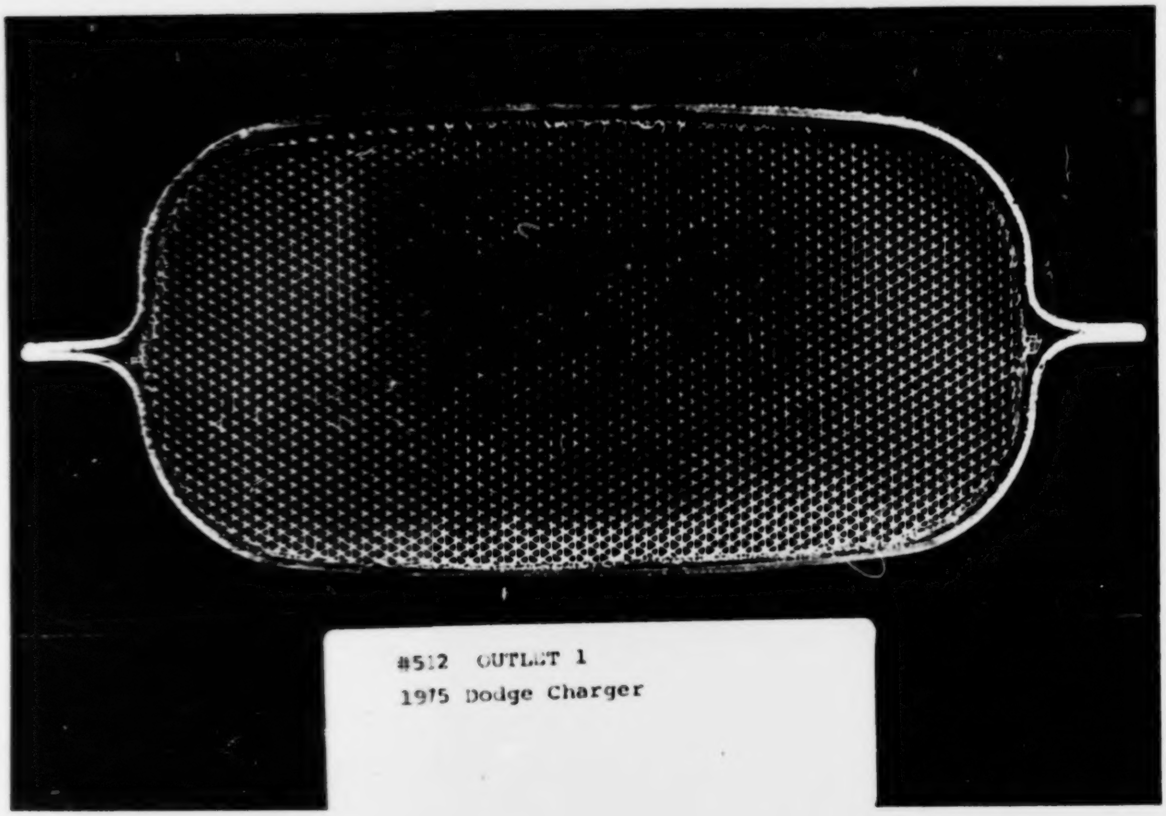


Photo 9. Converter #512 Outlet 1

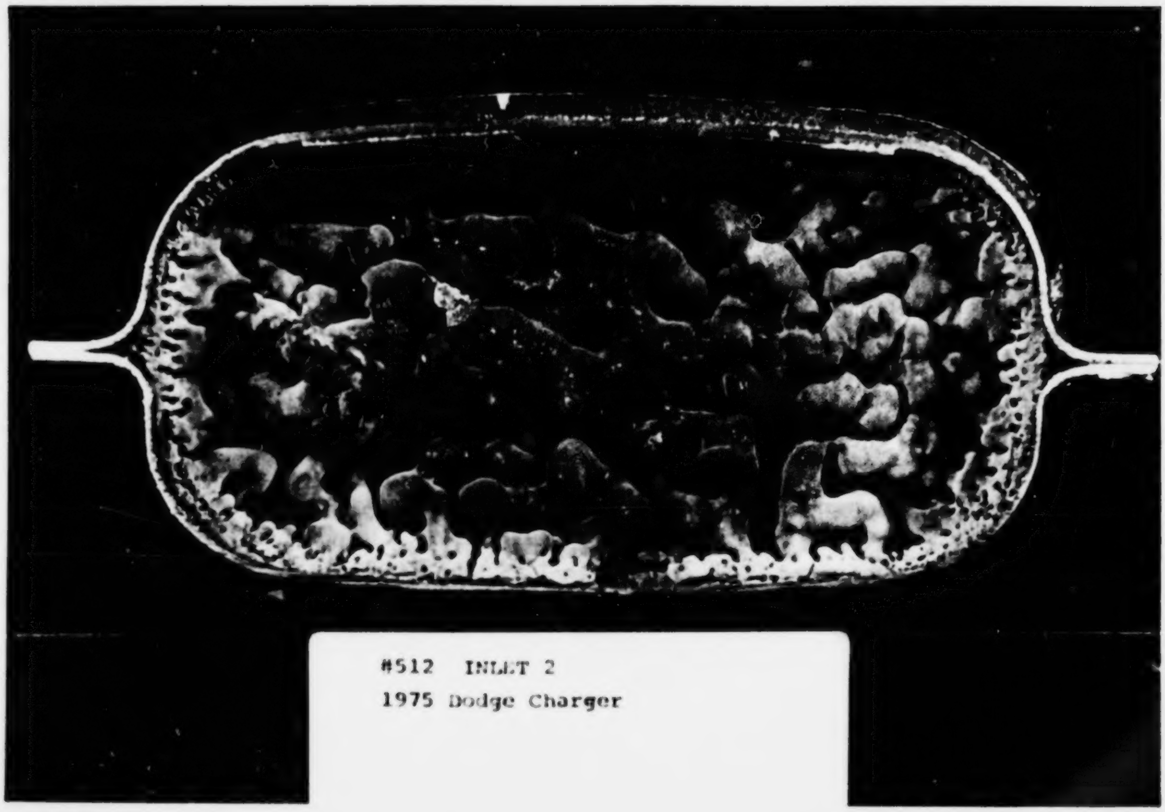


Photo 10. Converter #512 Inlet 2
(showing melt)

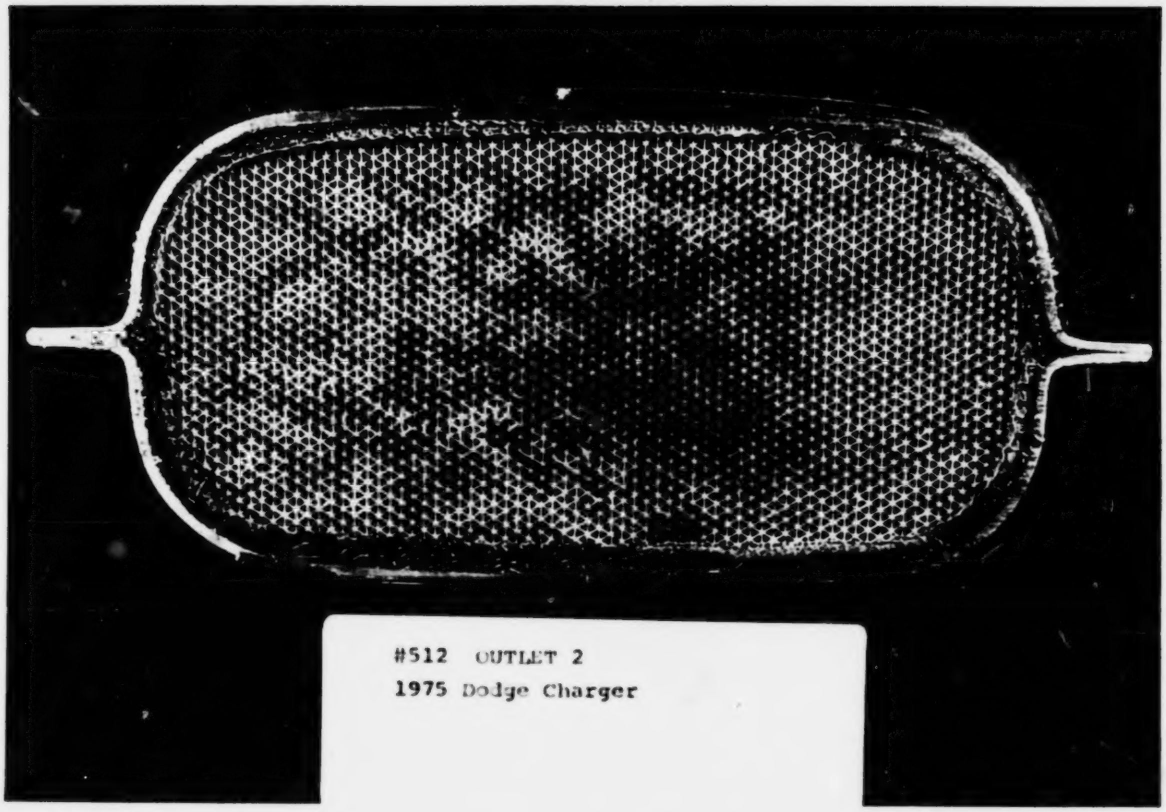


Photo 11. Converter #512 Outlet 2

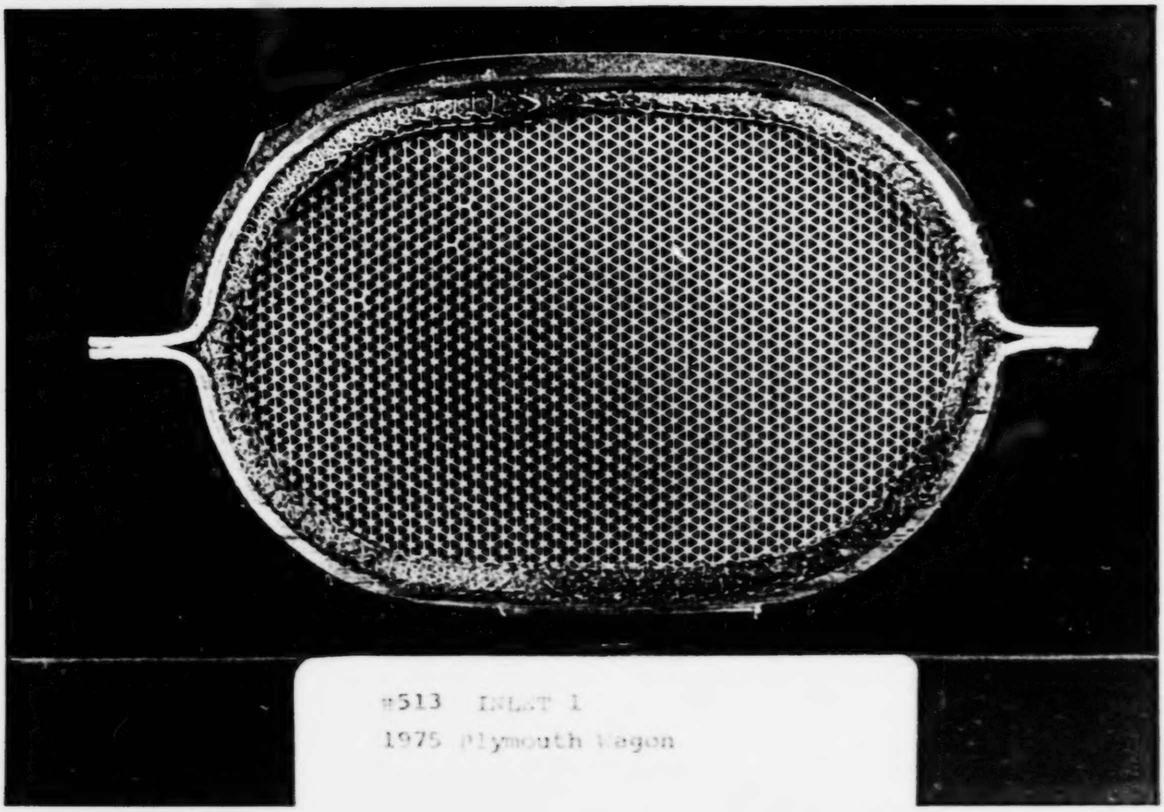


Photo 12. Converter #513 Inlet 1

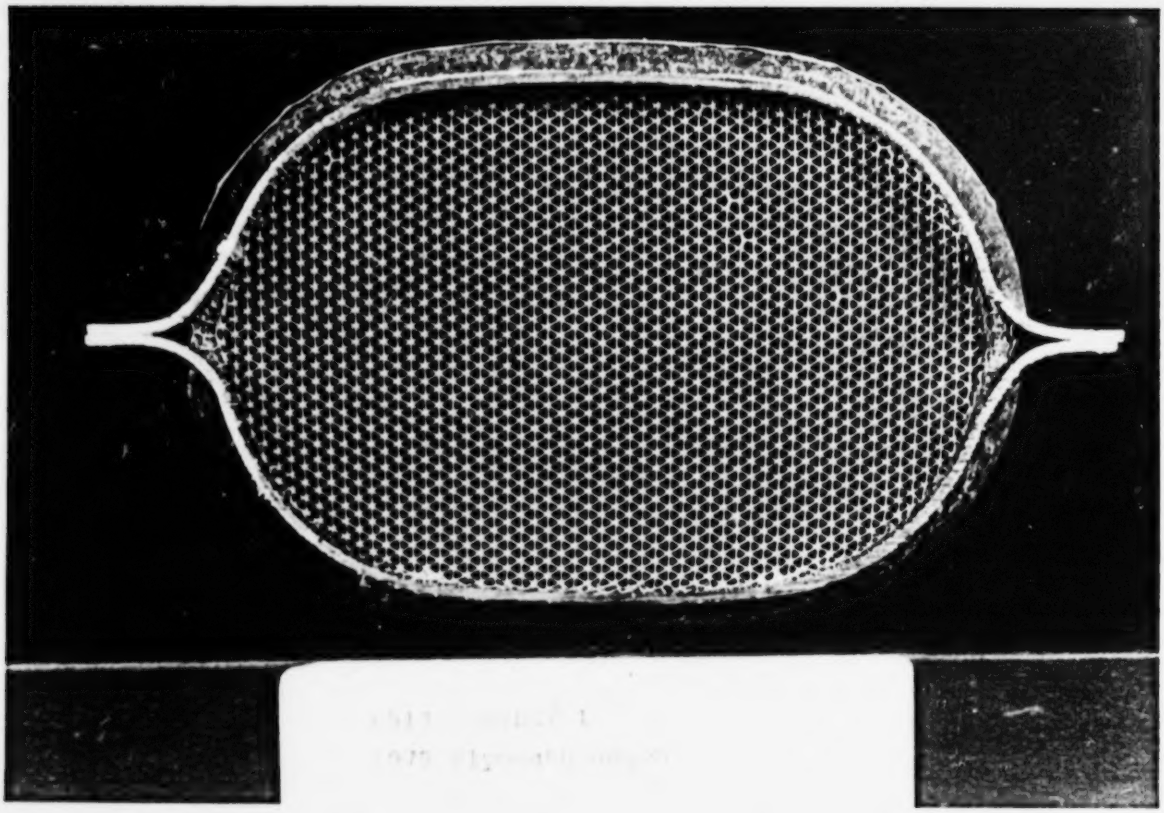


Photo 13. Converter #513 Outlet 1

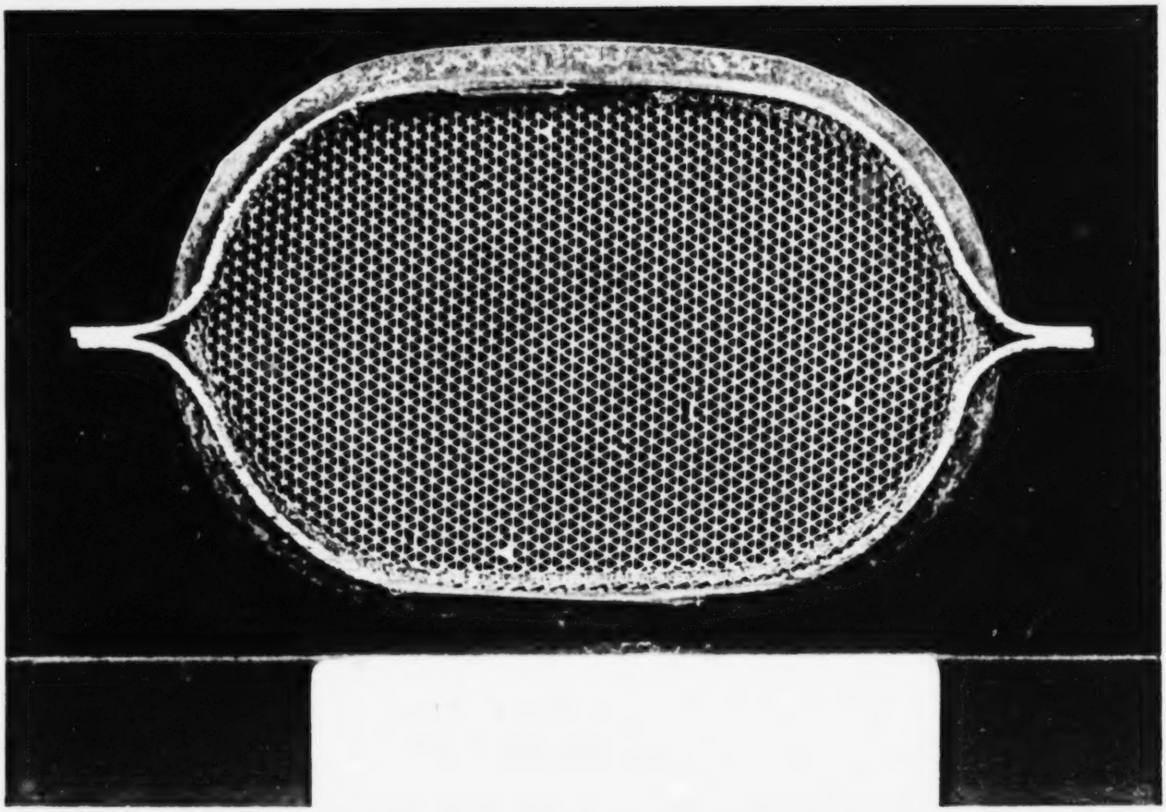


Photo 14. Converter #513 Inlet 2

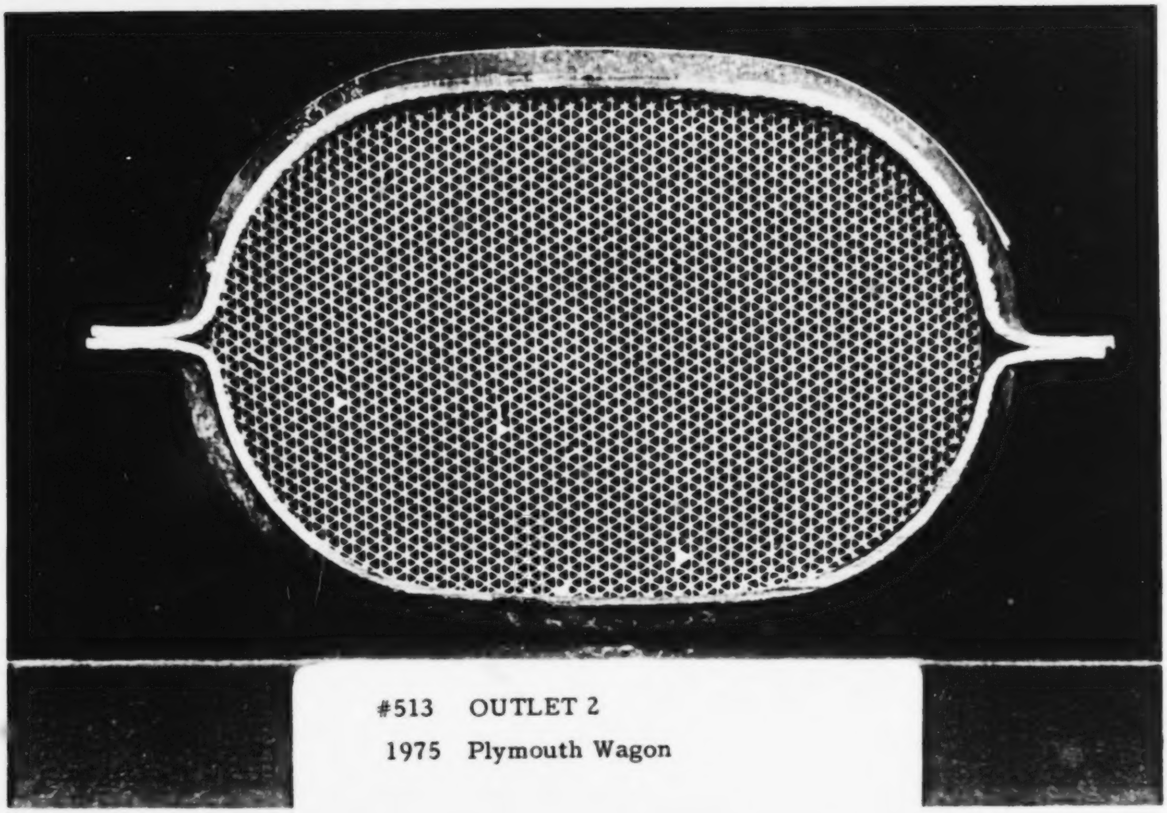


Photo 15. Converter #513 Outlet 2

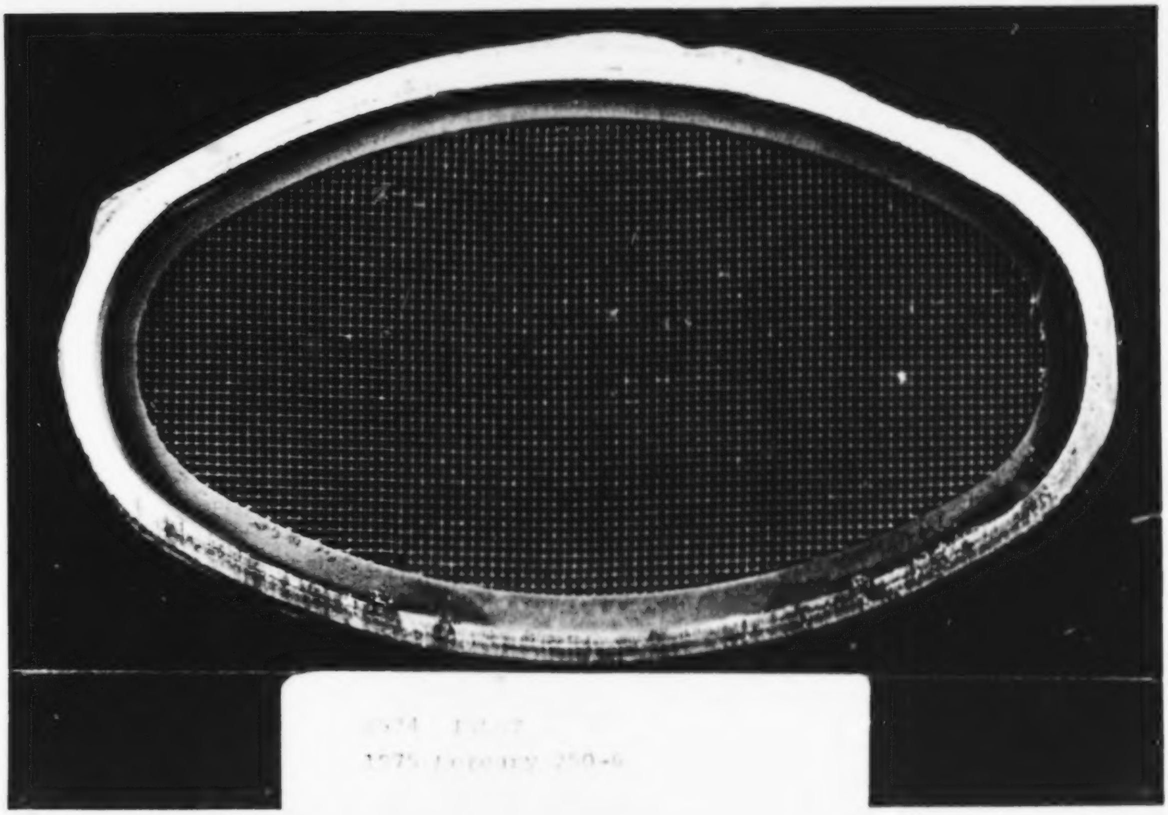


Photo 16. Converter #524 Inlet

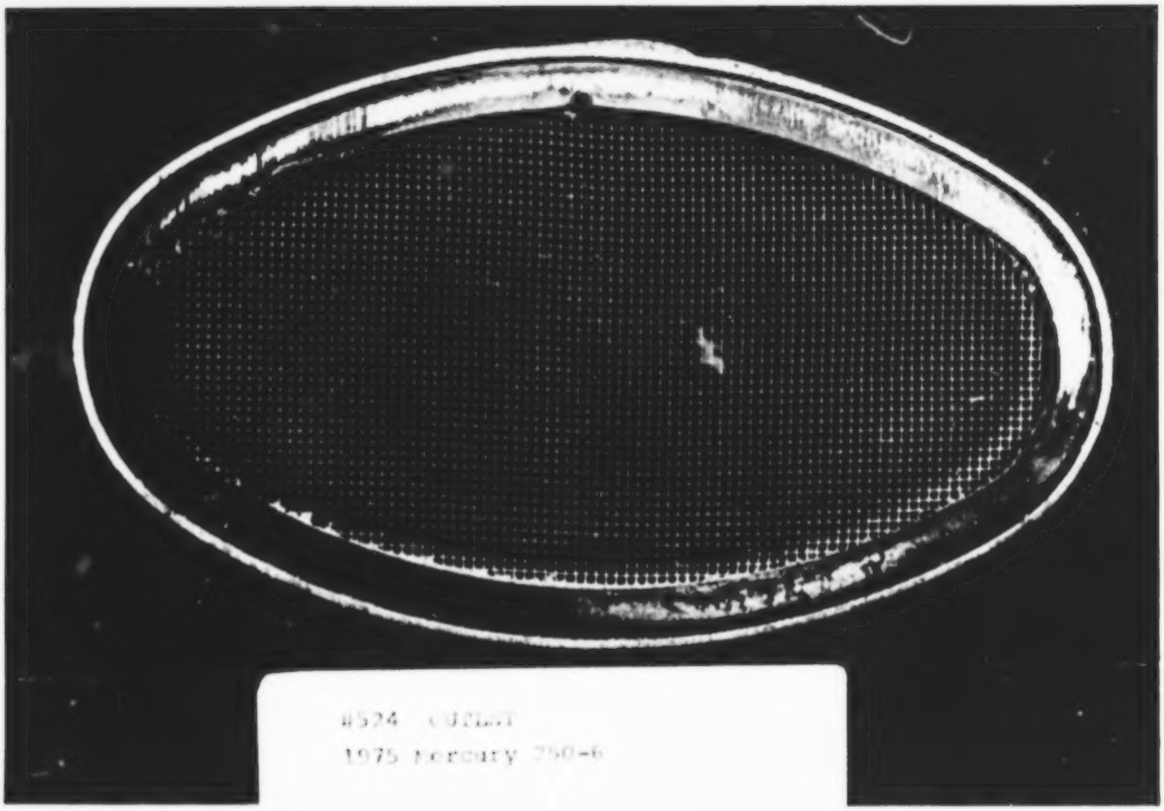


Photo 17. Converter #524 Outlet

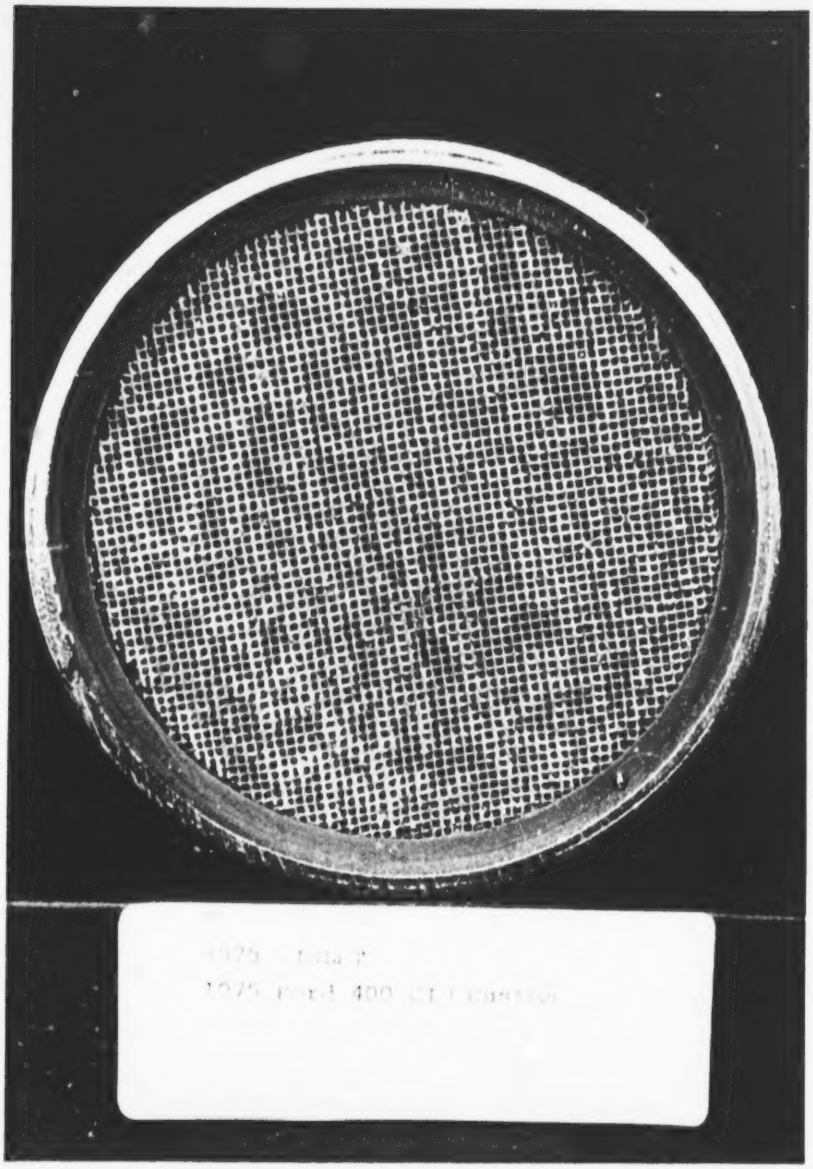


Photo 18. Converter #525 Inlet

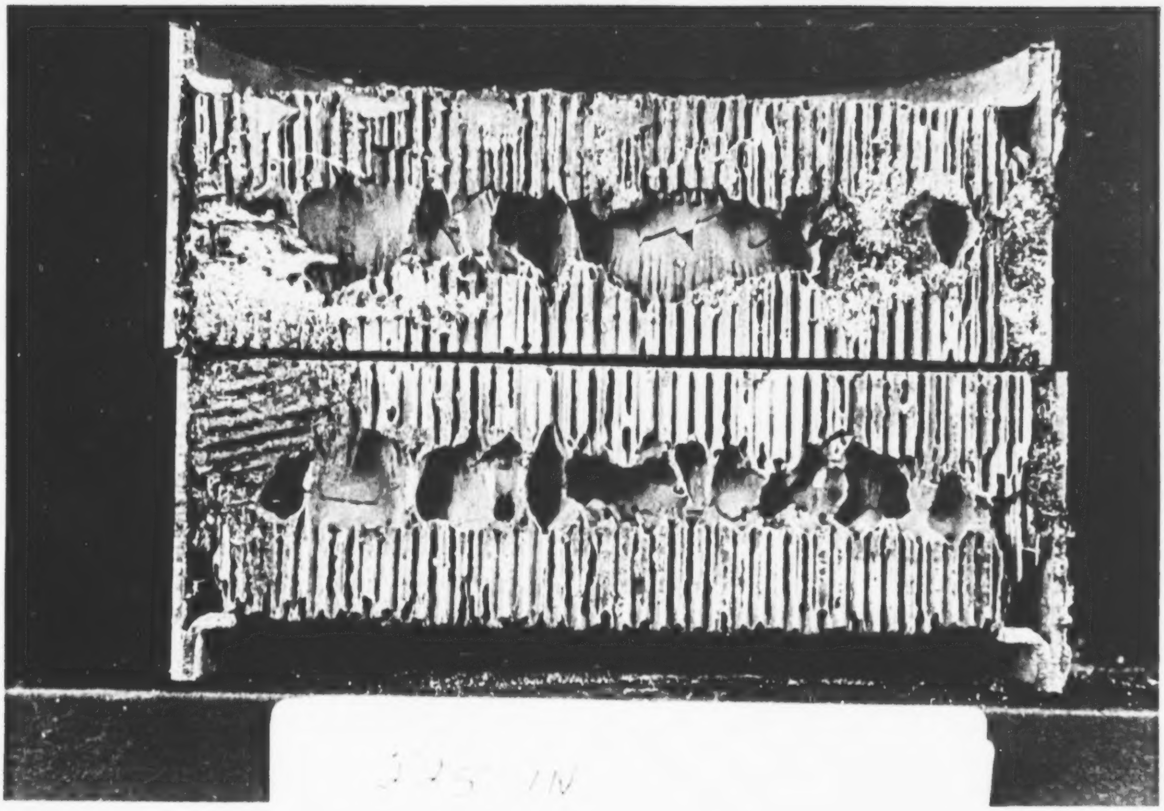


Photo 19. Converter #525 Inlet
(showing melt)

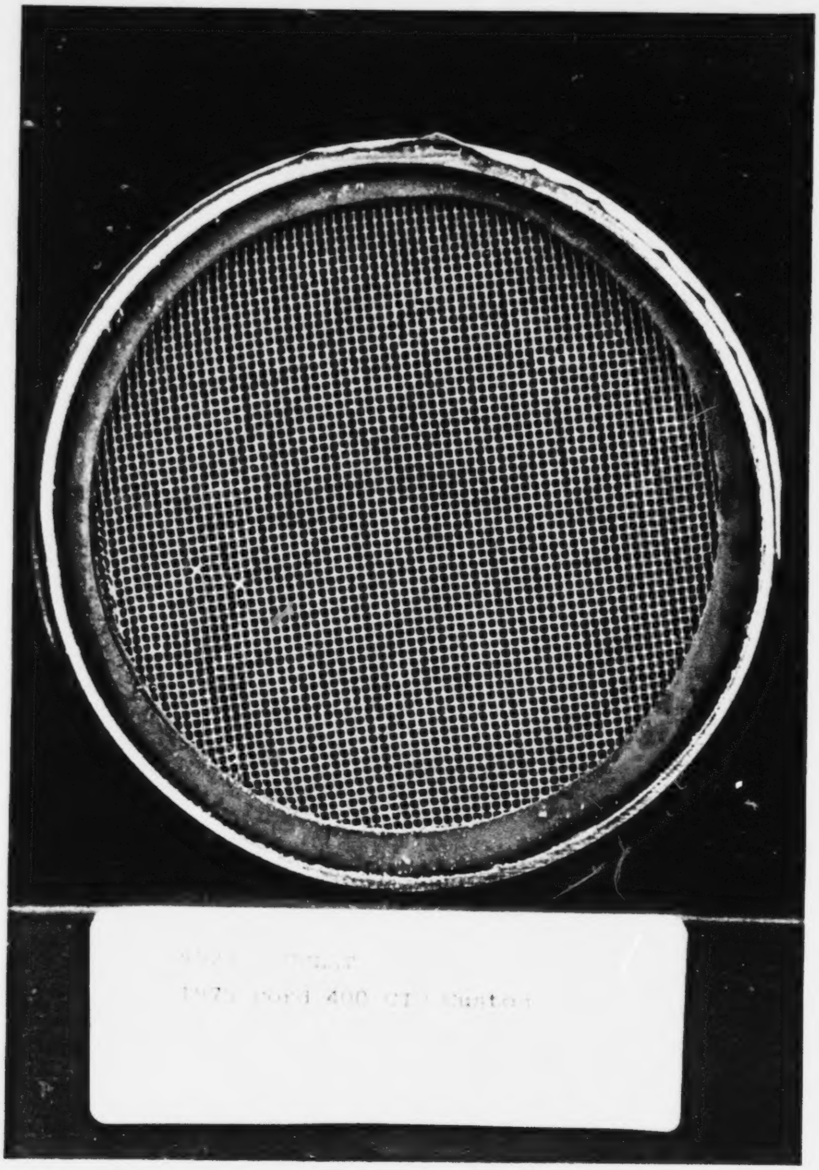


Photo 20. Converter #525 Outlet

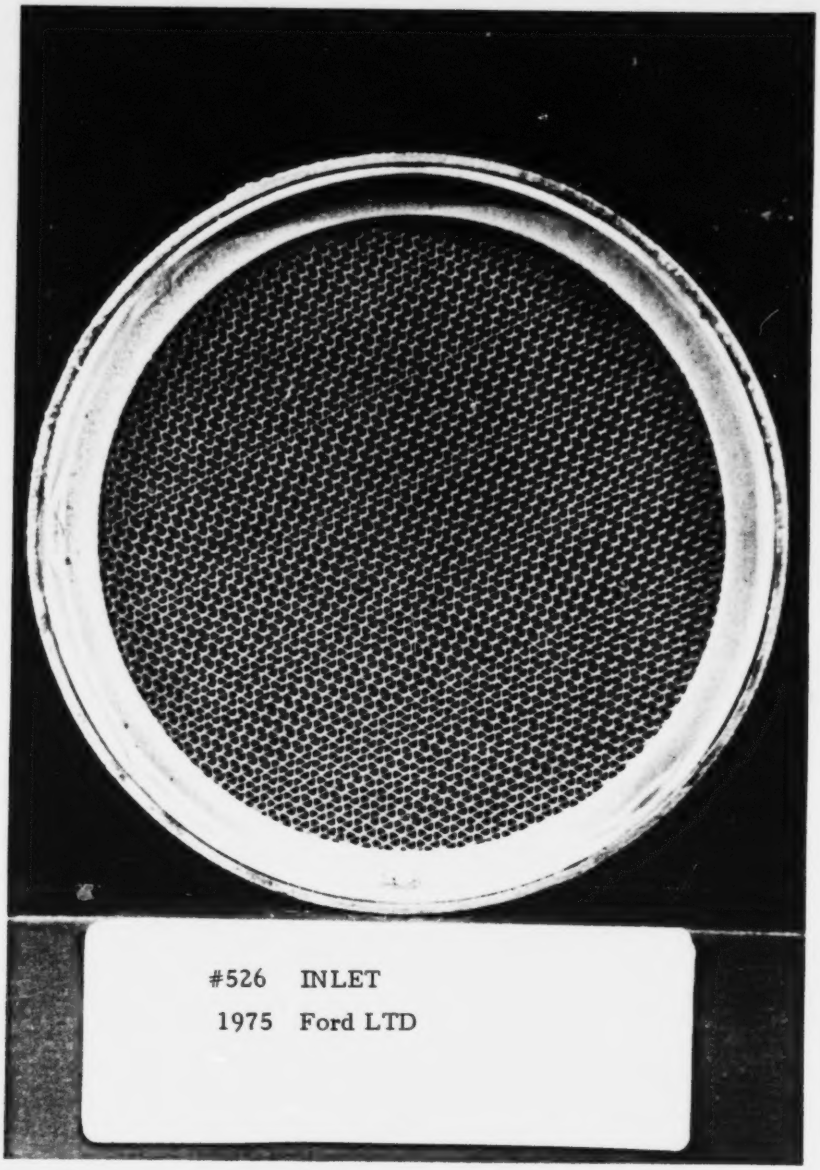


Photo 21. Converter #526 Inlet

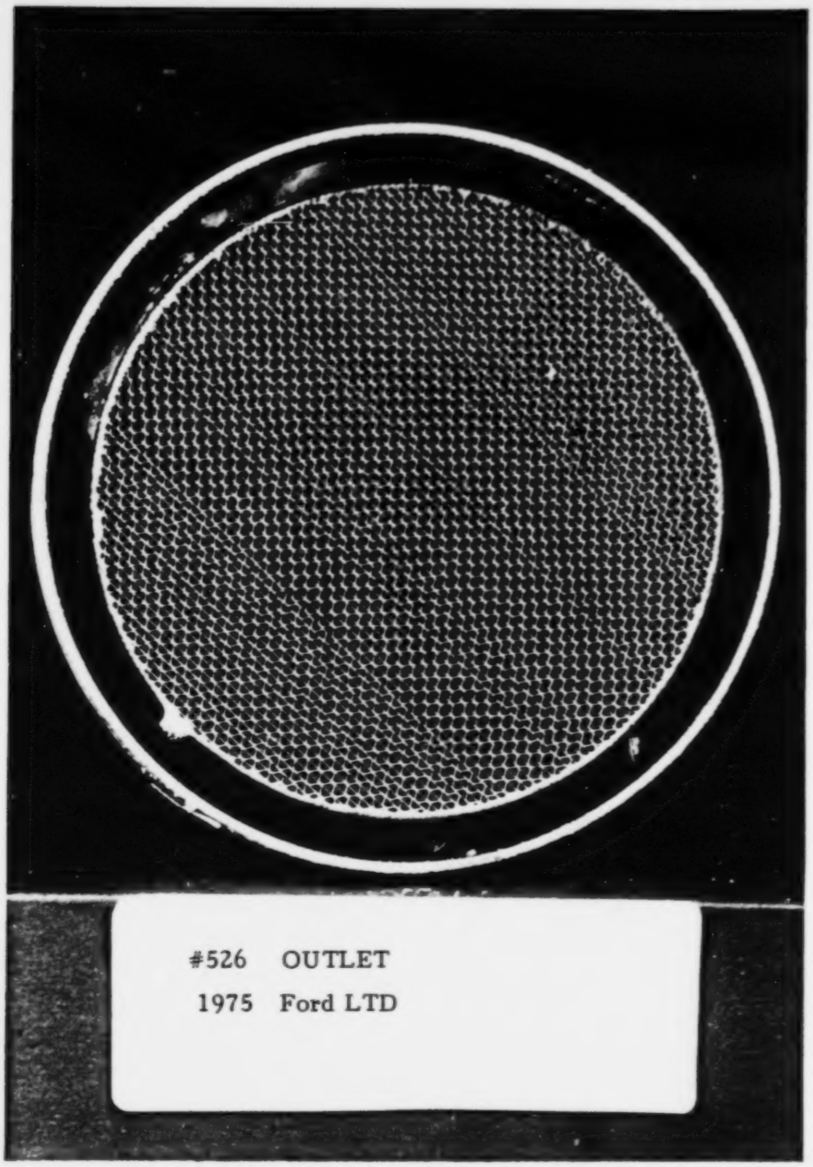


Photo 22. Converter #526 Outlet

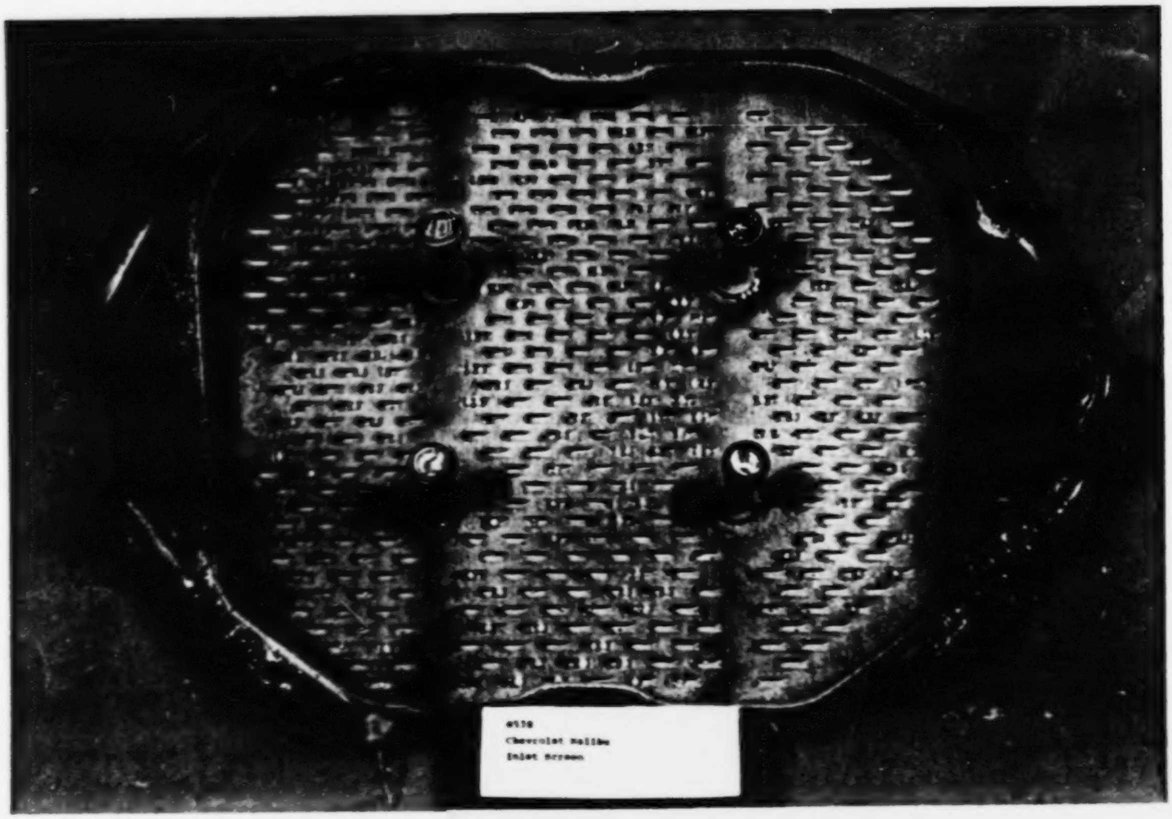


Photo 23. Converter #538 Inlet Screen

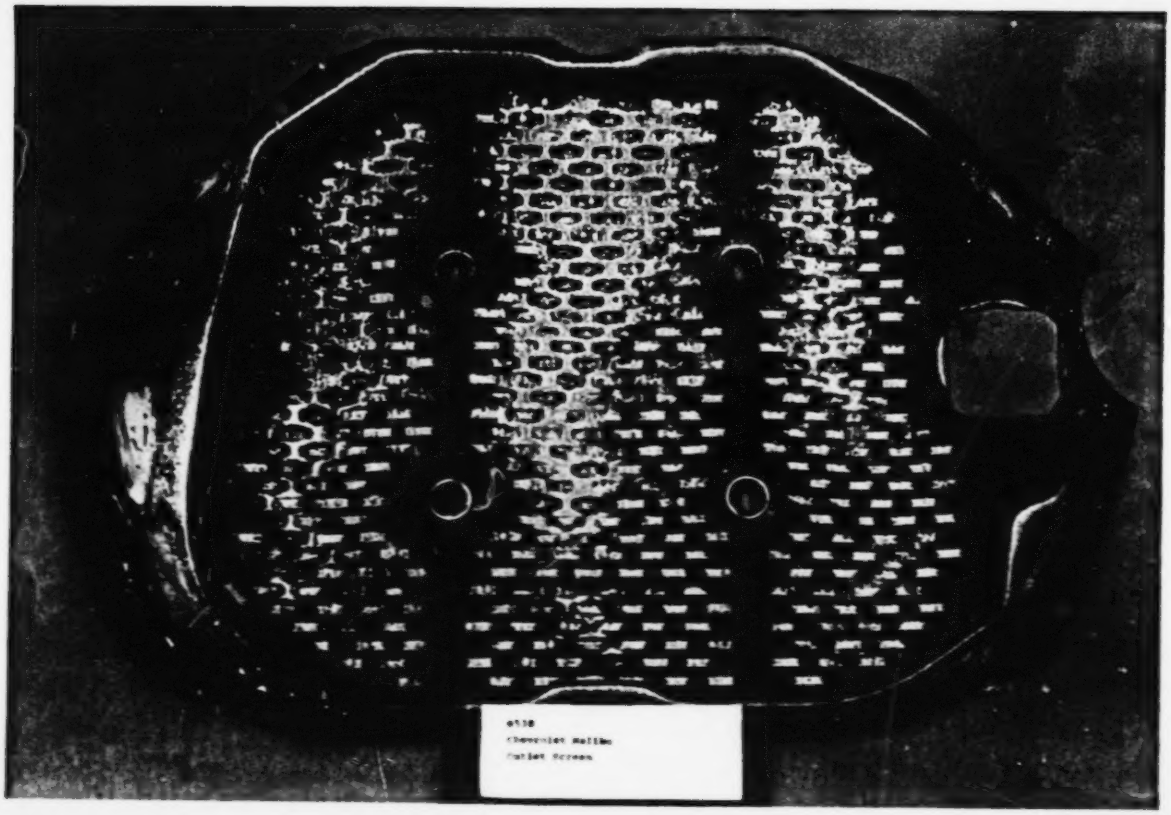


Photo 24. Converter #538
Front of Outlet Screen

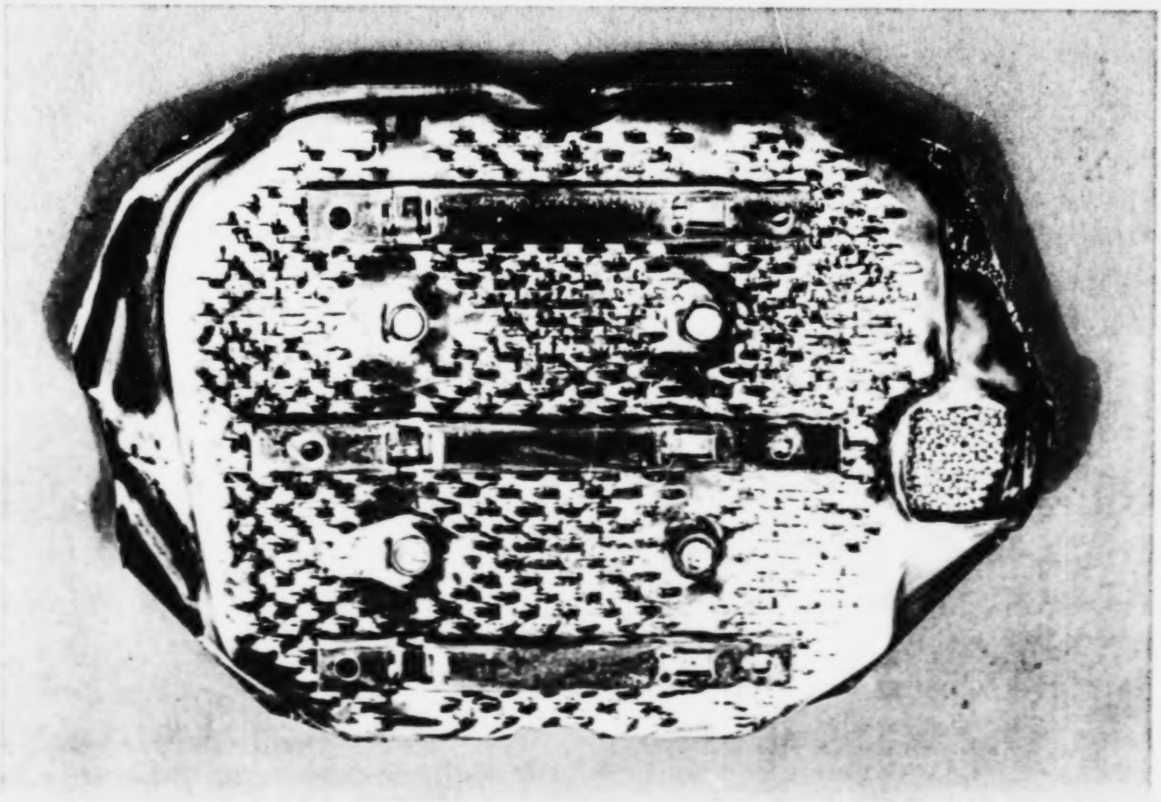


Photo 25. Converter #538
Back of Outlet Screen

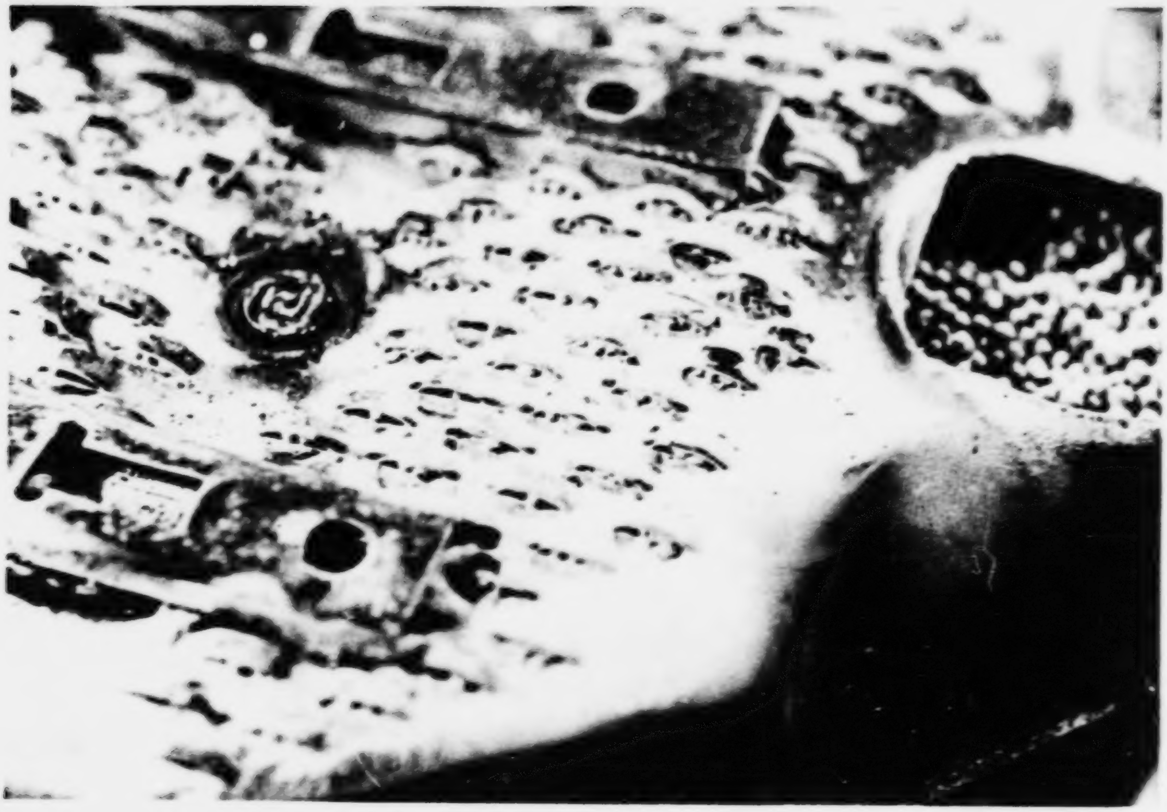


Photo 26. Converter #538
Back of Outlet Screen (Close-up)

SECTION II

CHARACTERIZATION OF CATALYST SAMPLES BY INSTRUMENTAL TECHNIQUES

Report by Leonard Niebylski

A. X-ray Diffraction Analyses
    1. Core Samples
        a. Chrysler Cars
        b. Ford Cars
        c. GM Cars
    2. Surface Deposits
        a. Chrysler Cars
        b. Ford Cars
        c. GM Cars
B. X-ray Energy Dispersive Spectroscopy - Surface and Core
C. Surface Area Measurements
D. Differential Thermal Analysis
E. X-ray Diffraction Pattern Charts
F. Specific Surface Area Data Tables
G. Differential Thermal Analysis Curves
H. Scanning Electron Micrographs

A. X-ray Diffraction Analyses

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Both core and surface deposits were analyzed by X-ray diffraction. A scanning X-ray diffractometer with filtered copper-X-radiation was used. The monolithic core with deposits was ground in an automated mortar and pestle apparatus. The ground material was placed on a microscope slide with doubled-sided scotch tape being used to secure the deposit. All materials were analyzed in duplicate. The extrudate and spherical pelleted catalysts were similarly processed with several pellets used as the composite sample. The surface deposits on both pellets and at the entrance and passages of the monolith were analyzed. In some instances, particularly when very little deposit was present, core material became intermixed with the surface deposits.

The X-ray diffraction patterns obtained for each of the catalysts provided are included with this report. Pattern analyses yielded no surprises with exception of catalyst 525 which had a melt down. An unknown crystalline material was characterized for the fused core material.

The reported crystalline composition of the monolith used as a support material as manufactured has been confirmed through our X-ray analysis of supports obtained directly from the manufacturer before wash coating and catalytic metal processing. The general composition of Corning Monoliths is

Cordierite, $2MgO2Al_2O_35SiO_2$ dominant phase

Varying trace amounts of other phases include:

Alumina	alpha-Al_2O_3
Mullite	$3Al_2O_32SiO_2$
Spinel	$MgAl_2O_4$

A wash coat is added to the surface of the monolith support. This material is either gamma or theta alumina or a mixture of the two. This high surface alumina is present in a few mils thickness. It is this layer of the support that become catalytically activated with platinum and palladium. Normally our X-ray diffractional analyses will not pick up this high surface alumina unless the coating is exceptionally thick and free of deposits of combustion.

Pelleted, spherical and extruded catalysts, are normally all pure gamma or theta, high surface area alumina. The average crystallite size of the alumina will generally range about 100 to 300 Angstroms. No wash coat is necessary for this material, therefore the platinum and palladium are added to the pelletized surface after the pellet is formed to conserve on catalytic metals used.

The specific core analyses for all the nine catalysts supplied is summarized in the following:

1. X-ray Diffraction Analyses Summary of Catalystic Supports

- Core Analysis Only -

a. Chrysler Cars: Two Corning monoliths were used in each of the cars. The monoliths have a triangular shaped open space with 236 cells per square inch. Some variations were noted in core composition of the two monoliths supplied.

Test 511-1	major component	cordierite
	~5%	α-Al_2O_3
	15-20%	α-SiO_2
Test 511-2	major component	cordierite
	~5%	α-Al_2O_3
Test 512-1 and 512-2	major component	cordierite
	5-10%	α-Al_2O_3
	15-20%	Unknown phase 297[1]
Test 513-1 and 513-2	major component	cordierite
	5%	α-Al_2O_3
	15-20%	Unknown 321[1]

NOTE: [1] Unknown phases are designed by the d-value of the strongest diffraction line. Different unknown number designations indicates a different crystalline phase is present.

b. Ford Cars: Catalysts from tests 524 and 525 used square hole, 200 cells per square inch Corning supports. The catalyst from test 526 used a rippled support of 256 cells per square inch manufactured by American Lava. This support material is no longer being manufactered. Two supports were used per car in this test series.

Test 524	major component	cordierite
	10%	$MgAl_2O_4$
	10-15%	α-Al_2O_3
	10%	mullite

Test 525 — A portion of this catalyst had a melt down. Both the fused and the non-fused ceramics were analyzed.

Non-fused Core:	major component	cordierite
	$\leq$ 5%	α-Al_2O_3
	5-10%	$MgAl_2O_4$
Fused Core:	40%	cordierite
	30-35%	Unknown 257[1]
	$<$ 10%	α-Al_2O_3
	$<$ 10%	mullite

Test 526	major component 20% 25-30% $<$10%	cordierite $MgAl_2O_4$ α-Al_2O_3 mullite

c. General Motors Cars: Pelleted support materials are used in these test catalysts. Test 537 used extruded, cylindrical shaped pellets about 1/8 inch long - 3/32 inches diameter. Catalytic supports from test 538 and 539 are spherical with the spheres ranging from 3/32 to 5/32 inches in diameter.

Test 537	50% 50%	alpha alumina theta alumina

The presence of alpha alumina suggests the catalyst was subjected to an elevated temperature in excess of 1800°F during a portion of its operation.

Test 538	40% 60% trace	gamma alumina theta alumina of either eta or delta Al_2O_3
Test 539	60% 40% trace	gamma alumina theta alumina delta alumina

2. - Surface Deposit Analysis -

The core material composition has been subtracted out from the diffraction pattern if present. The composition data is thst of materials deposited on the catalyst support which is formed by combustion or volitilization of materials from the engine. The deposits from pelleted catalyst is that material which is on the pellet surface. The monolith surface deposit is that which is deposited at the entrance to the monolith passages.

a. Chrysler Cars:

Test 511-1 and 511-2	70-75% 15-20% remainier	$PbSO_4$ Mn_3O_4 unknown
Test 512-1 and 512-2	85% 15%	$PbSO_4$ $PbOPbSO_4$
Test 513-1 and 513-2	15-20% 75%	$PbSO_4$ Mixture of unknowns

b. Ford Cars:

In all instances one or more unknown phases are present in the surface deposits. Catalysts from tests 524 and 526 are approximately the same. Deposits from car test 525 contains a different set of unknowns with the exception of possibly one phase being common in all deposits. (See Diffraction Patterns)

c. General Motors Cars:

Unlike the Ford test samples, the surface deposits on General Motor pelleted catalysts are poorly developed crystallites. Mn_3O_4 is present in concentrations up to 20% in all catalysts samples. The unknowns present on catalysts of Ford cars are also present on these supports. (See diffraction patterns).

B Surface Analysis by X-ray Energy Dispersive Spectroscopy

To help characterize the unknown compositions of the surface deposits observed by X-ray diffraction on Ford and General Motors catalysts a surface analysis was performed using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The type of surface deposits formed on catalyst surfaces is illustrated in the SEM micrographs for catalyst from tests, 512 (Chrysler monolith) 525 (Ford monolith) and 537 (GM extrudate) and 538 (GM deposits on pelleted catalysis will vary from 10 to 40 microns. The deposits within the channels of a monolith will vary from 15 to 60 microns and at the entrance to the monoliths deposits may be more than 100 microns thick.

Energy dispersive spectroscopy is a technique by which electrons generating an image in the SEM also generate X-rays characteristic of the element being irradiated. These X-ray radiations are picked up and a surface analysis of a 10-20 microns layer thick is obtained. In areas where the deposit may be thinner than 20 microns some of the supporting catalysts will be detected.

All nine catalysts were analyzed by EDS. The data are presented in the table on the following page. Only the 9 major elements generating emissions are detected. These data are quantized relative to weight present on the surface at the location being irradiated. Elements in greater than 10% concentration will have a significant influence on the bulk crystalline composition of the deposits.

It should be pointed that Mg, Al and Si are due primarily to support materials. The presence of Pb and Mn is due to an antiknock present in gasoline the vehicle used. The presence of Zn, Ca, P and S are due to oil additive constituents. The EDS technique being used does not readily distinguish between sulfur and lead in concentration less than 5%. Consequently when sulfur or lead is detected there will be a question as to which element is present. Another surface analytical technique is needed to establish the absolute quantitative concentrations of sulfur and lead. When the concentration of Pb is greater than 5% secondary lead lines are detected therefore the concentration of lead present is absolute. Unfortunately it does not yield sulfur data unless sulfur is present in excess of 5%.

Based on the surface analysis data it appears that lead contamination occurred in about half the catalysts provided.

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X-ray Energy Dispersive Spectroscopy Analysis of Automotive Exhaust Catalysts from Chrysler Vehicles

(powdered = entire catalyst used as sample, flat = surface deposit only)

```
ID:SAMPLE #511/ POWDERED
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               6.399
   AL              45.632
   SI              32.610
   P                6.265
   PB               4.578
   CA               0.918
   TI               0.373
   MN               2.354
   ZN               0.680
```

```
ID:DEPOSITS ON END OF S #511
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               1.357
   AL               6.957
   SI               3.180
   P               29.074
   CA               7.168
   MN               9.958
   ZN               1.885
   PB              34.699
   FE               5.509
```

```
ID:FLAT OF SAMPLE #511
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               1.177
   AL               4.495
   P               37.707
   CA              10.331
   MN              26.824
   ZN               5.770
   SI               0.849
   PB              12.650
```

```
ID:SAMPLE #512/ POWDERED
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               3.463
   AL              21.631
   SI              19.107
   P                1.124
   CA               0.426
   TI               0.263
   MN               1.051
   ZN               0.368
   PB              52.549
```

```
ID:DEPOSITS ON END OF S #512
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               2.362
   AL               0.962
   SI               0.478
   P               12.027
   CA               5.120
   MN              45.100
   ZN               5.788
   PB              28.128
```

```
ID:FLAT OF SAMPLE #512
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               2.290
   AL               3.809
   SI               1.568
   P                1.821
   CA               1.965
   MN              11.915
   ZN               3.949
   PB              72.513
```

```
ID:SAMPLE #513/ POWDERED
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               6.357
   AL              54.594
   SI              32.135
   P                1.724
   PB               1.479
   CA               0.638
   TI               0.629
   MN               1.914
   ZN               0.311
```

```
ID:FLAT OF SAMPLE #513
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               0.582
   AL              34.333
   P               28.855
   CA               6.625
   MN              21.755
   ZN               3.472
   SI               0.352
   PB               3.157
   BA               0.673
```

```
ID:FLAT OF SAMPLE #513
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG               1.055
   AL              29.205
   P               29.228
   CA               7.363
   MN              24.275
   ZN               3.556
   SI               0.733
   PB               3.874
   BA               0.765
```

X-ray Energy Dispersive Spectroscopy Analysis of Automotive Exhaust Catalysts from Ford Vehicles

(powdered = entire catalyst used as sample, flat = surface deposit only)

```
ID:SAMPLE #524/ POWDERED
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               5.033
  AL              42.387
  SI              29.104
  P                3.813
  PB               8.369
  CA               0.597
  MN               8.515
  ZN               0.523
  CE               1.558
```

```
ID:FLAT OF SAMPLE #524
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               1.575
  AL               9.565
  P                6.347
  CA               1.348
  MN              63.924
  ZN               1.654
  SI               6.305
  PB               9.069
```

```
ID:FLAT OF SAMPLE #524
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               1.178
  AL               8.303
  P                6.595
  CA               1.223
  MN              66.057
  ZN               1.873
  SI               5.454
  PB               9.098
```

```
ID:SAMPLE #525/ POWDERED
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               6.615
  AL              33.373
  SI              32.162
  P                8.029
  PB               3.756
  CA               5.072
  TI               0.655
  MN               6.629
  ZN               3.591
```

```
ID:FLAT OF SAMPLE #525
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               3.146
  AL               9.925
  P               15.179
  CA               6.958
  MN              48.699
  ZN               5.701
  SI               8.872
  PB               1.183
  TI               0.279
```

```
ID:FLAT OF SAMPLE #525
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               2.702
  AL               7.576
  P               22.229
  CA               8.112
  MN              45.547
  ZN               6.230
  SI               4.610
  PB               1.761
  TI               0.152
```

```
ID:SAMPLE #526/ POWDERED
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               1.707
  AL              53.612
  SI              26.123
  P                3.031
  PB               5.244
  K                2.221
  CE               2.423
  MN               4.994
  ZN               0.549
```

```
ID:SAMPLE #526/ POWDERED
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               1.707
  AL              53.612
  SI              26.123
  P                3.031
  PB               5.244
  K                2.221
  CE               2.423
  MN               4.994
  ZN               0.549
```

```
ID:FLAT OF SAMPLE #526
SEMIQ:NORMALIZE

ELEMENTS              %
  MG               1.078
  AL               9.503
  P               21.801
  CA               1.652
  MN              43.841
  ZN               3.054
  SI               3.019
  PB              14.594
```

X-ray Energy Dispersive Spectroscopy Analysis of Automotive Exhaust Catalysts from General Motors

(powdered = entire pellet used as sample)

```
ID:SAMPLE #537/ POWDERED
SEMIQ:NORMALIZE

 ELEMENTS             %
   AL               99.054
   SI                0.000
   P                 0.045
   CA                0.198
   MN                0.219
   FE                0.122
   ZN                0.003
   S/Pb              0.516
```

```
ID:PELLET SURFACE; SAMPLE #537
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG                0.000
   AL               66.886
   P                10.041
   CA                1.659
   MN               21.568
   ZN                1.360
   SI                0.249
   PB                2.023
```

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```
ID:SAMPLE #538/ POWDERED
SEMIQ:NORMALIZE

 ELEMENTS             %
   AL               98.191
   SI                0.000
   P                 0.448
   CA                0.268
   MN                0.213
   FE                0.117
   ZN                0.124
   PB                0.439
```

```
ID:PELLET SURFACE; SAMPLE #538
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG                3.978
   AL                5.131
   P                37.555
   CA               14.626
   MN               32.361
   ZN                5.373
   SI                0.352
   PB                0.387
```

```
ID:PELLET SURFACE; SAMPLE #538
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG                4.294
   AL                7.237
   P                39.149
   CA               13.464
   MN               29.613
   ZN                6.124
```

```
ID:SAMPLE #539/ POWDERED
SEMIQ:NORMALIZE

 ELEMENTS             %
   AL               97.699
   SI                0.000
   P                 0.535
   CA                0.410
   MN                0.486
   FE                0.269
   ZN                0.135
   PB                0.458
```

```
ID:PELLET SURFACE; SAMPLE #539
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG                1.308
   AL                7.209
   P                33.697
   CA               12.296
   MN               40.814
   ZN                3.751
   SI                0.000
   PB                0.735
```

```
ID:PELLET SURFACE; SAMPLE #539
SEMIQ:NORMALIZE

 ELEMENTS             %
   MG                1.168
   AL                8.248
   P                33.333
   CA               11.851
   MN               40.671
   ZN                3.735
   SI                0.000
   PB                0.803
```

Major Lead Contamination in Surface Deposits of Exhaust Catalysts

Chrysler		Ford		General Motors	
Test	Pb-Contamination	Test	Pb-Contamination	Test	Pb-Contamination
511	yes	524	yes	537	possibly some
512	yes	525	questionable	538	no
513	slight	526	yes	539	no

The unknowns in test 524 and 526 appear to be associated with manganese and lead. Whereas the unknowns detected in deposits on catalysts from test 537, 538, and 539 appear to be associated with Mn, Ca and P.

C. Surface Area Measurements

The catalytic activity of a material can be estimated by the specific surface area measured for that material. Multi-point B.E.T. analyses were obtained on all nine catalytic samples. An Accusorb Physical Adsorption Analyzer using nitrogen gas was used to provide the gas adsorption data. The specific surface area obtained for each sample is summarized in the table below. All the adsorption data are also provided in the attached computer print-out sheets.

Specific Surface Area of Test Catalysts

Sample Identification	Specific Surface Area (m^2/g)
511-1 Plymouth	8.68
512 Dodge	6.35
513-1 Plymouth	6.98
524 Ford	5.09
525 Ford 400	3.94
526 Ford LTD	8.08
537 GM	60.9
538 GM	108.0
539 GM	58.5

Pelletized catalysts normally rate 100 to 125 m^2/g when fresh. The lower readings of 58 and 60 m^2/g suggest some deactivation of the catalyst. Monolith support surface area measurements are deceiving since only a very thin wash coat provides the required high surface area for a catalytically active material. Therefore, its weight is insignificant compared to that of the monolith support itself. Consequently, surface area measurement of monoliths yield values of 5 to 10 m^2/g. Lower reading would suggest possibly some catalyst deactivation has occurred.

D. Differential Thermal Analysis

The solid state thermal properties of the monolith with its deposits were analyzed by differential thermal analysis (DTA). A DuPont 990 thermal analysis system was used. It is capable of measuring thermal changes up to 1200°C in a controlled atmosphere environment. The thermographs were obtained on all nine catalyst samples. Approximately 30 milligrams of sample which was ground and packed into a platinum cell. The atmosphere gas contained 0.8% oxygen in nitrogen. The oxygen level closely approximates the oxygen level in exhaust stream when A/F ratio is at stoichiometric and no added air is injected into the exhaust stream. Both heating and cooling curves were obtained and registered on the same chart. The DTA thermograms are provided with this report. The thermal sensitivity of the thermal responses obtained was set at two levels, 0.2 and 1.0 millicalories per inch scale deflection. Under these sensitivity conditions the thermogram provides a view of the major and minor thermal reactions that are taking place. The system was heated at a rate of 10°C per minute. A similar cooling rate was programed. The thermal data obtained on the nine samples is summarized below:

Chrysler Catalysts: Tests 511, 512, and 513, Corning's 236 c/in^2 supports: A minor, but sharp exotherm, occurs between 170-190°C which cannot be associated with any known physical change except surface desorption.

A pronounced oxidation reaction reaches an exothermic peak at:

400°C for catalyst 511

260°C for catalyst 512

360°C for catalyst 513

In all the samples a minor exothermic reaction appears to be initiated between 700-750°C. This appears as increased noise in the thermal recording. It is believed these reactions are due to sintering or surface reactions.

A subtle, minor endothermic reaction appears to range from about 620°C to 850°C. This change is undoubtedly due to a crystallographic change that occurs within the core.

A sharp exothermic phase transition occurs in catalyst 513, at 695°C this is a phase reversal transition since it also occurs endothermically on cooling.

Ford Catalysts

Test samples 524 and 525 represent Corning 200 cells/in^2 monoliths. The monolith from test 526, represents an American Lava support which is considerably less pure than the Corning samples. These were analyzed by differential thermal analysis. Some slight high temperature differences are noted in the DTA curves that would distinguish the Lava support from the Corning support.

An initial minor but sharp exotherm occurs at 170-190°C similar to that observed with the Chrysler supports. Oxidation reactions occur with the exotherm reaching a maximum at:

370°C for test 524

340°C for test 525

425°C for test 526

In the range of 650°-700° an endotherm is initiated and extends to 900°C. This thermal reaction is believed to be due to a crystallographic phase change.

Minor phase transitions provide a sharp exotherm in the cooling curves in the range of 970-1030°C. The higher transition temperature represents a Lava support.

General Motors Catalysts: These pelleted catalysts were analyzed similar to the monolith supports. Catalyst pellets from test 537 were extrudates, and pellets from tests 538 and 539 were spheres. Thermal differences between the two forms was slight, only the high temperature phase transitions can distinguish between the two forms. The spheres show a sharp exotherm on cooling at 1035°C while the extrudate produces a exotherm at 960°C phase transitions. These transitions are reversible on heating and cooling.

Overall the pelleted catalysts produce an initial strong endotherm in the range of 35 to 80°C, possibly adsorbed water or gas is being desorbed. A minor exotherm is observed in the range of 170 to 190°C similar to that noted with the monoliths, although it is less pronounced in the case of the pellets.

An oxidation maximum is noted for the different tests samples at:

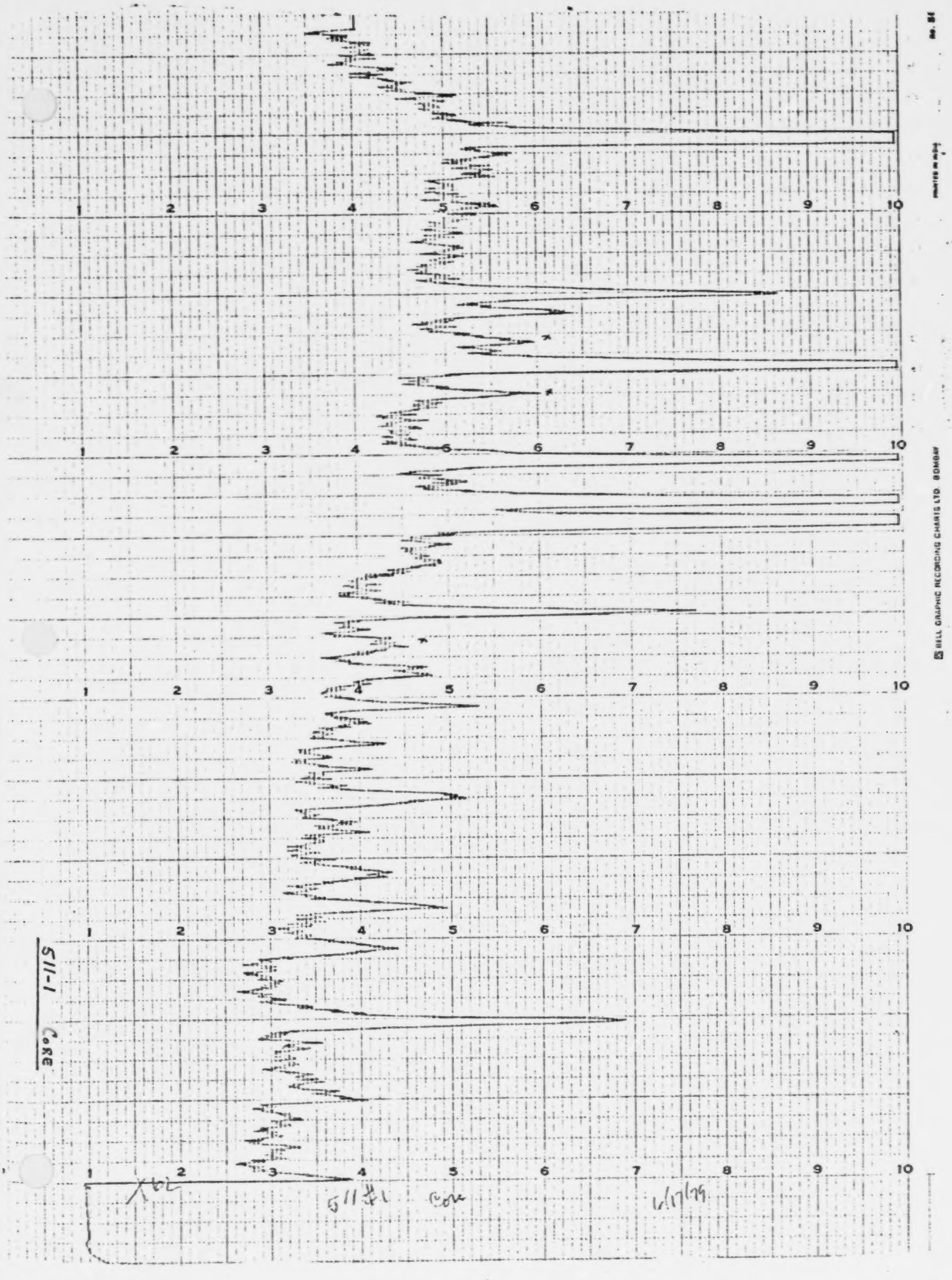
350°C for test sample 537

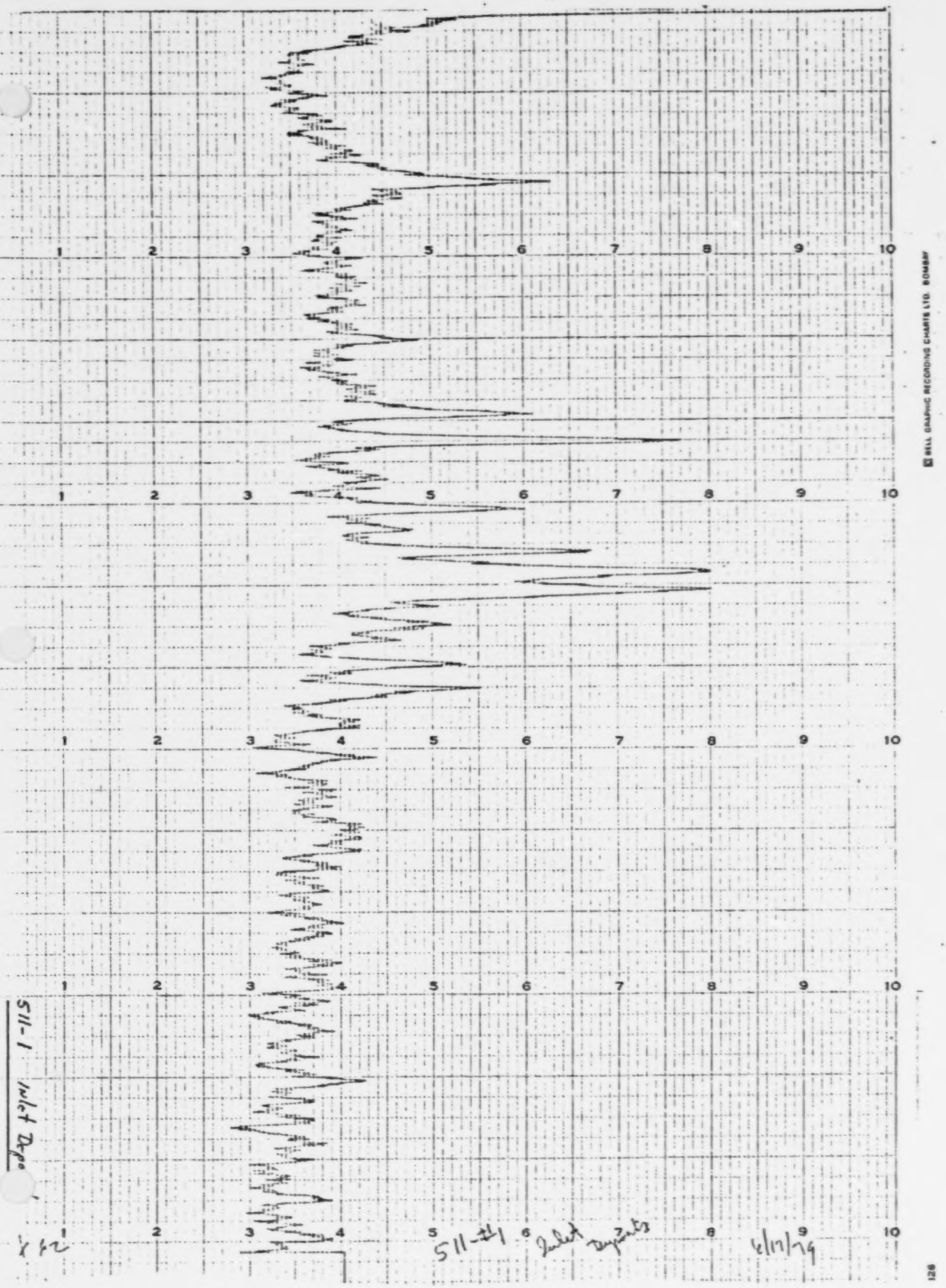
290°C for test sample 538

310°C for test sample 539

In conclusion, it would be necessary to combine high temperature X-ray diffraction analysis and thermogravometric analysis to gas chromatographic analysis to ascertain the subtle thermal changes that are being observed. In general though the reactions are slight and do not affect the overall physical structure of the support when heated to 1200°C. Possibly higher temperatures may be required to see a catastrophic change in structure.

E. X-RAY DIFFRACTION PATTERNS

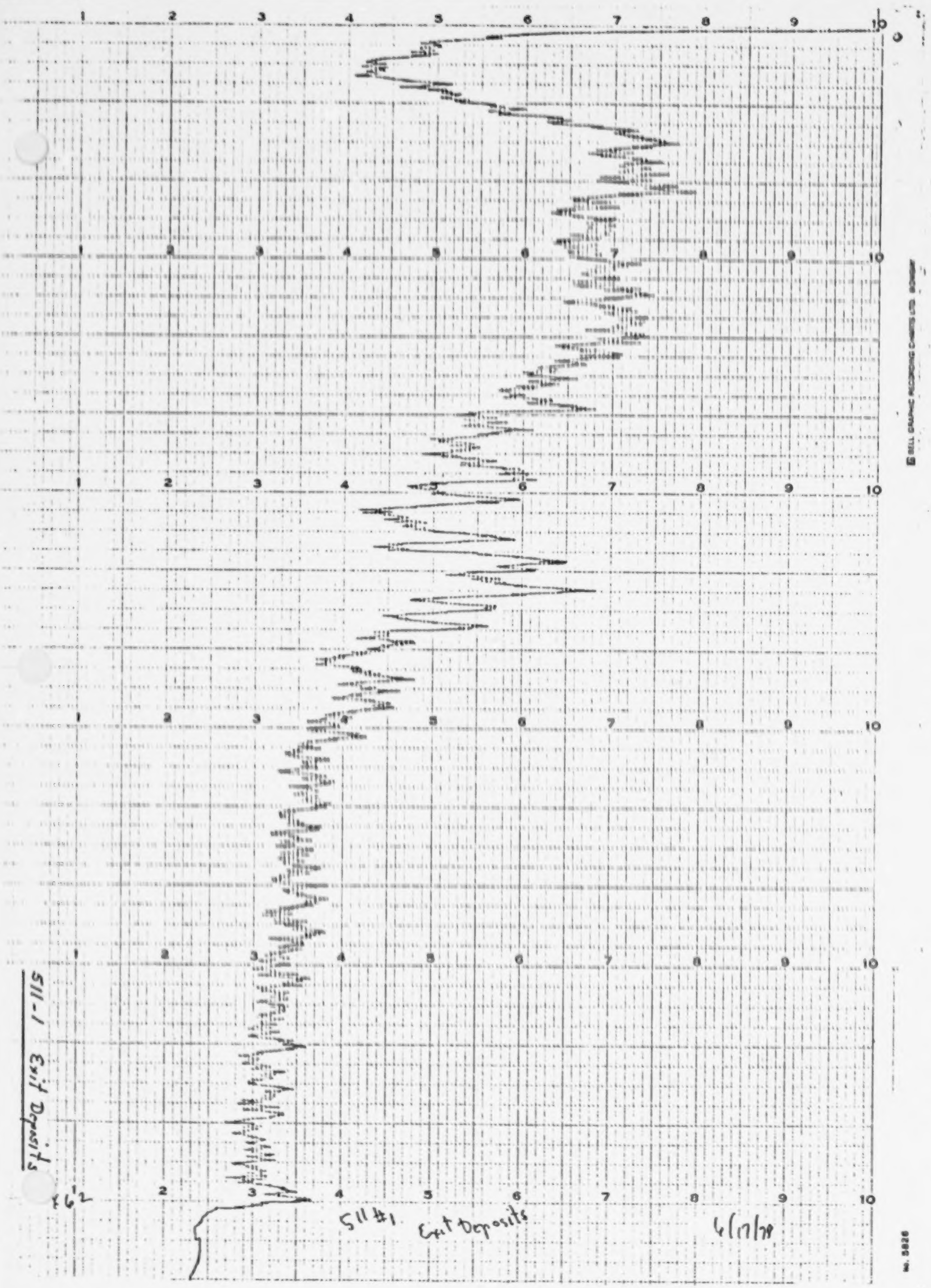

1 2 3 4 5 6 7 8 9 10
511-1 Core
XRD
511 #1 Core
1/17/79
BELL GRAPHIC RECORDING CHARTS LTD. BOMBAY

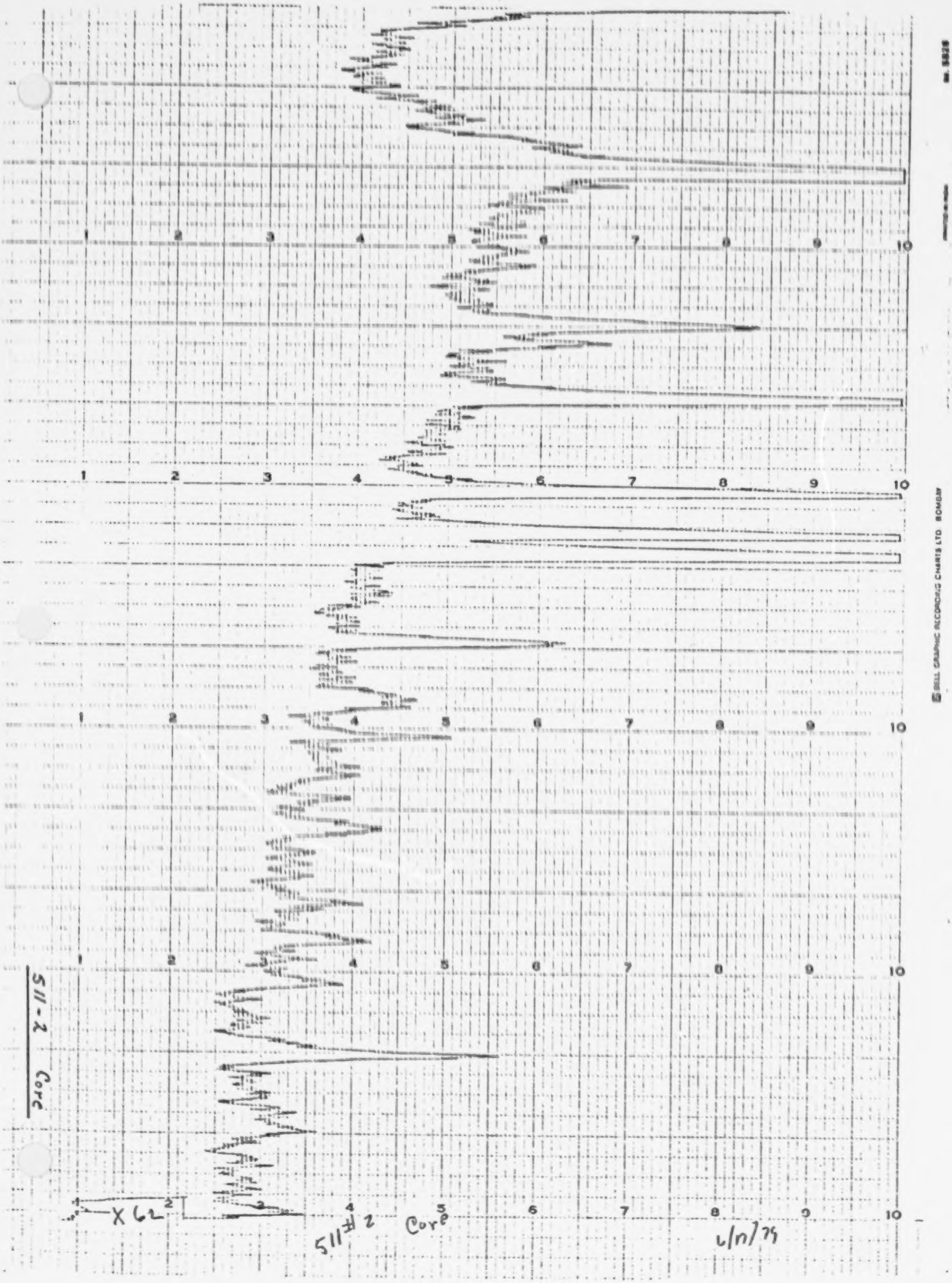


511-1 Inlet Depo

X f2

511-#1 Inlet Deposits 6/17/79

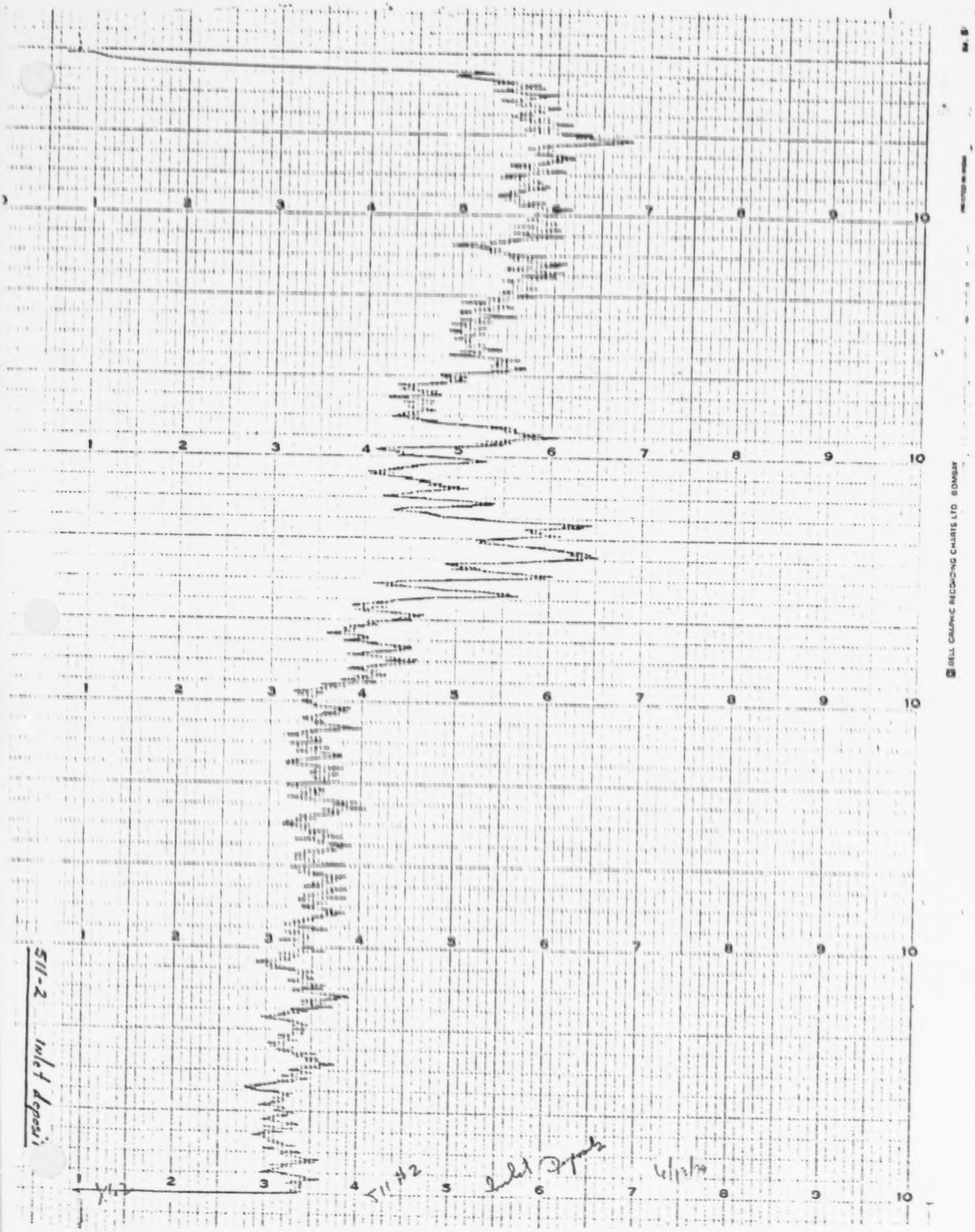

1 2 3 4 5 6 7 8 9 10
511-1 Exit Deposits
x6
511 #1 Exit Deposits
6/17/78
BELL GRAPHIC RECORDING CHARTS LTD
5826



511-2 Core

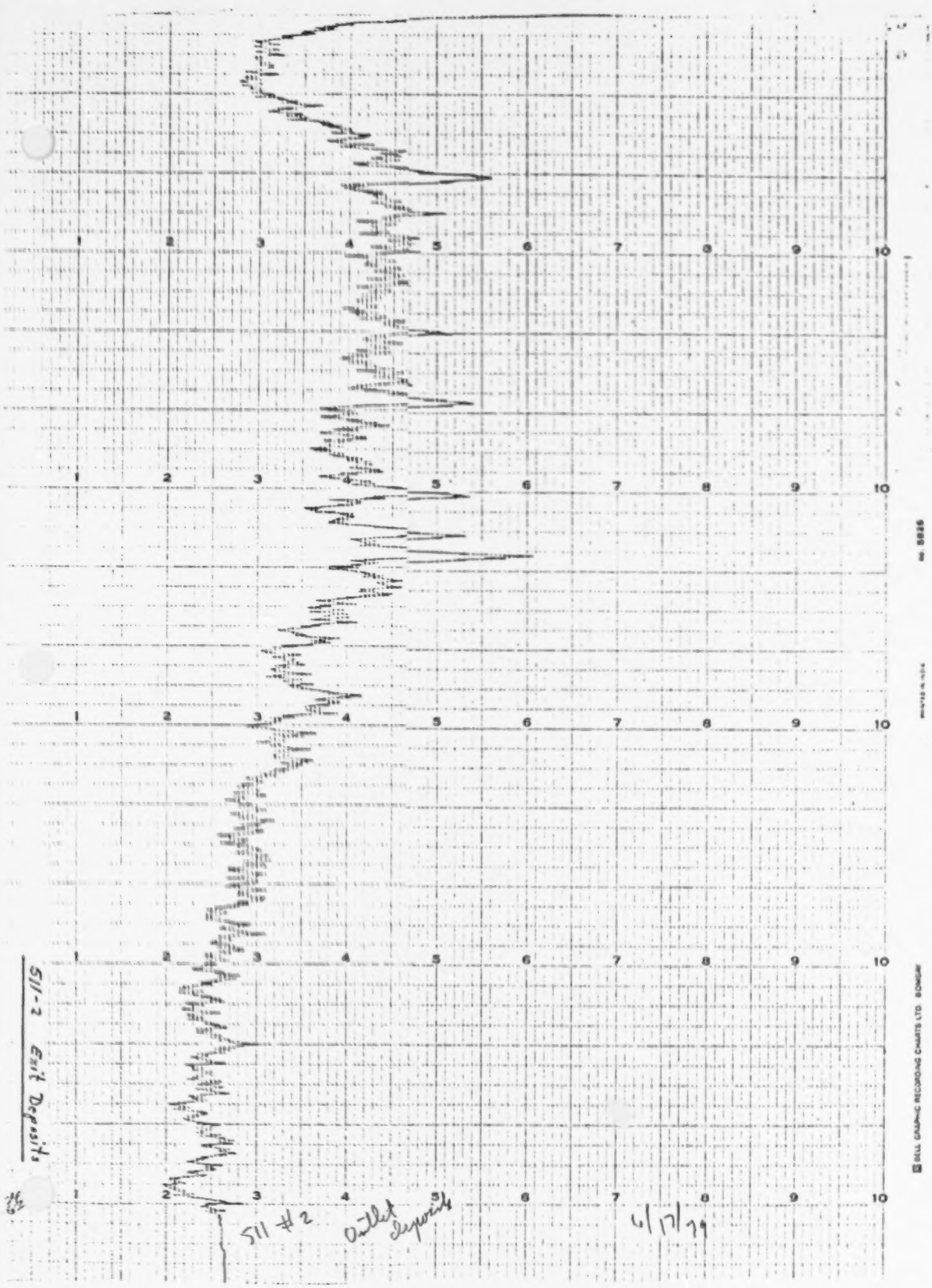
511 #2 Core

6/11/79



511-2 Inlet deposi

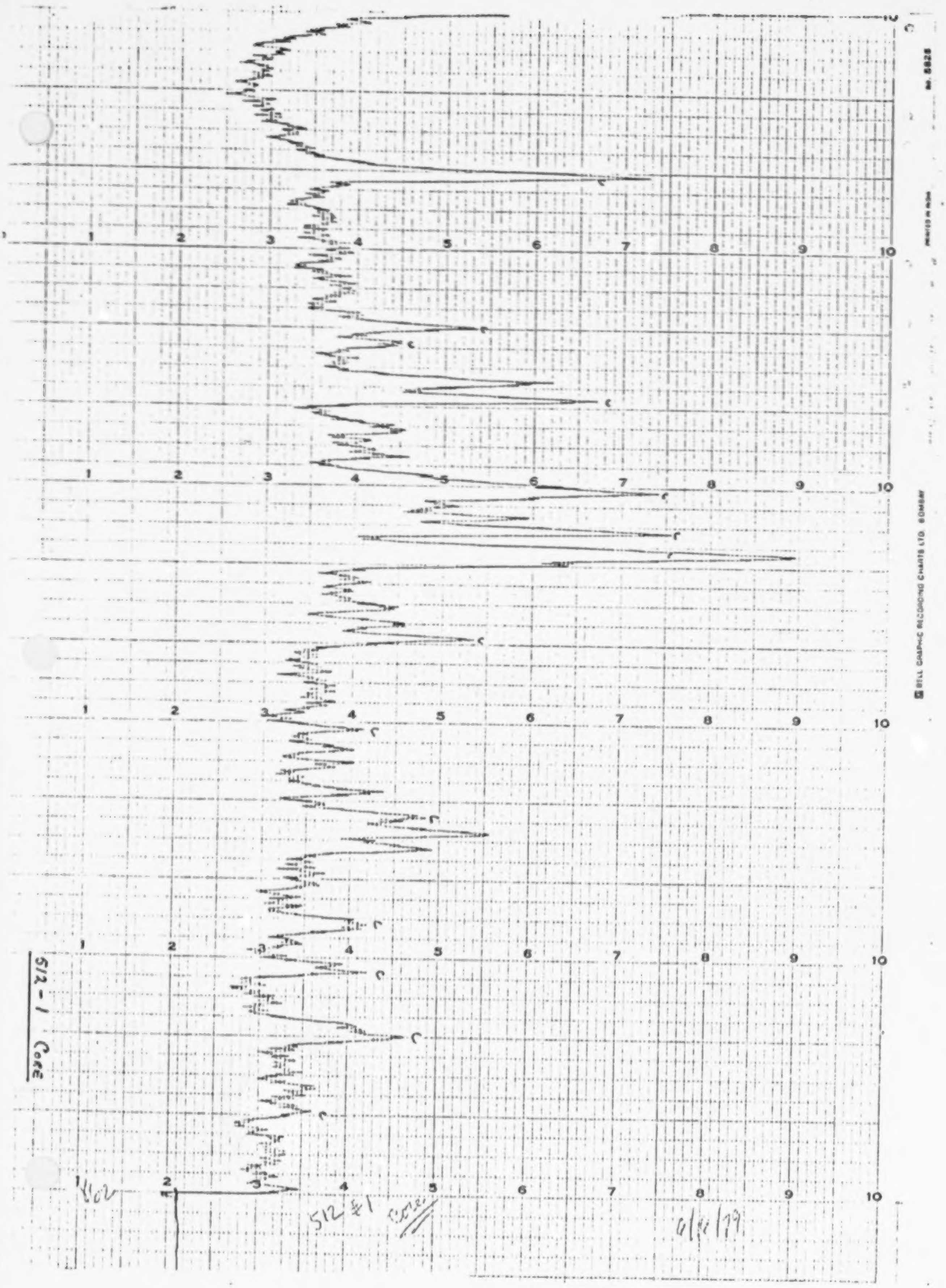
511 #2 Inlet Deposits 4/13/79



511-2 Exit Deposits

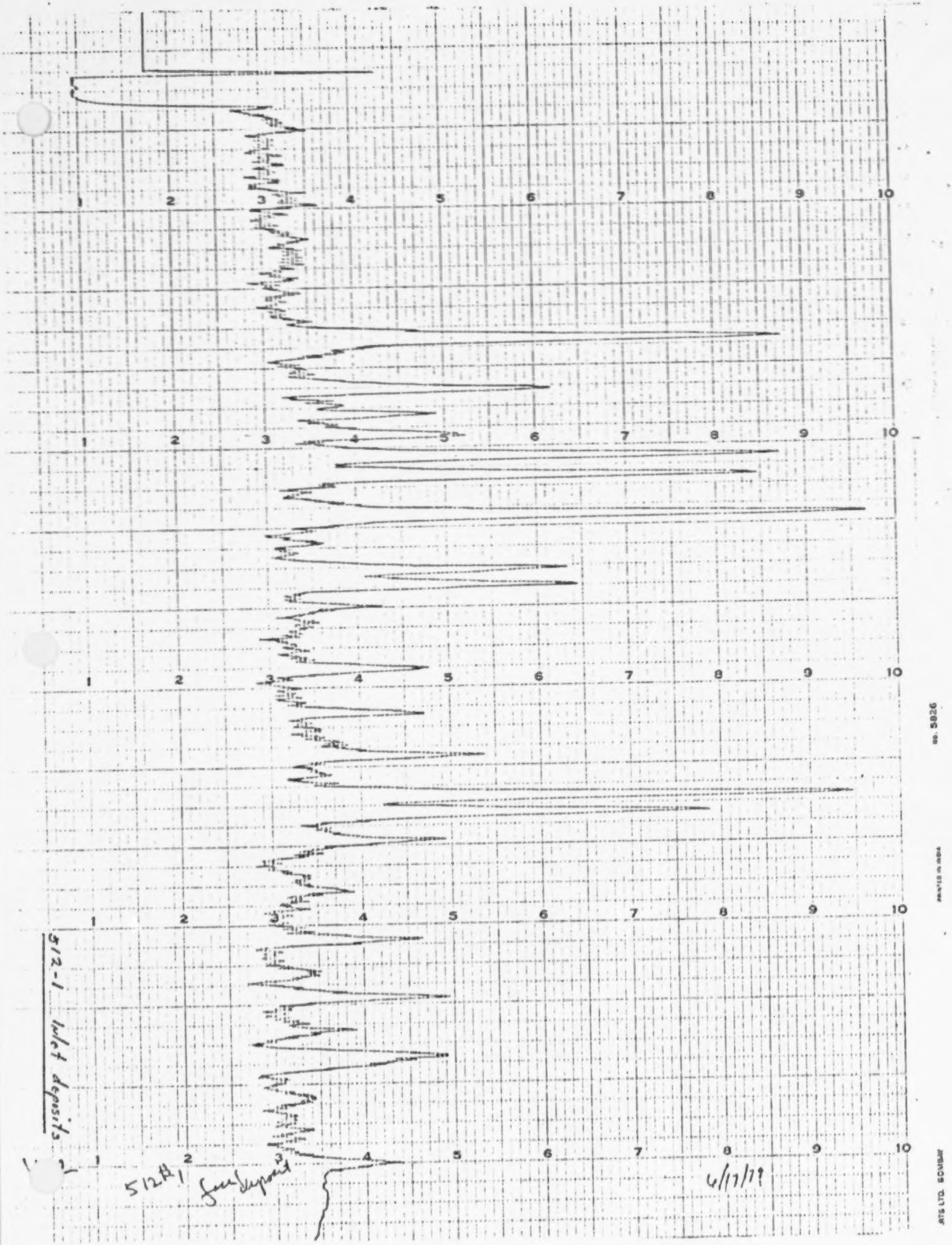
511 #2 Outlet deposits

6/17/79



512 #1 core

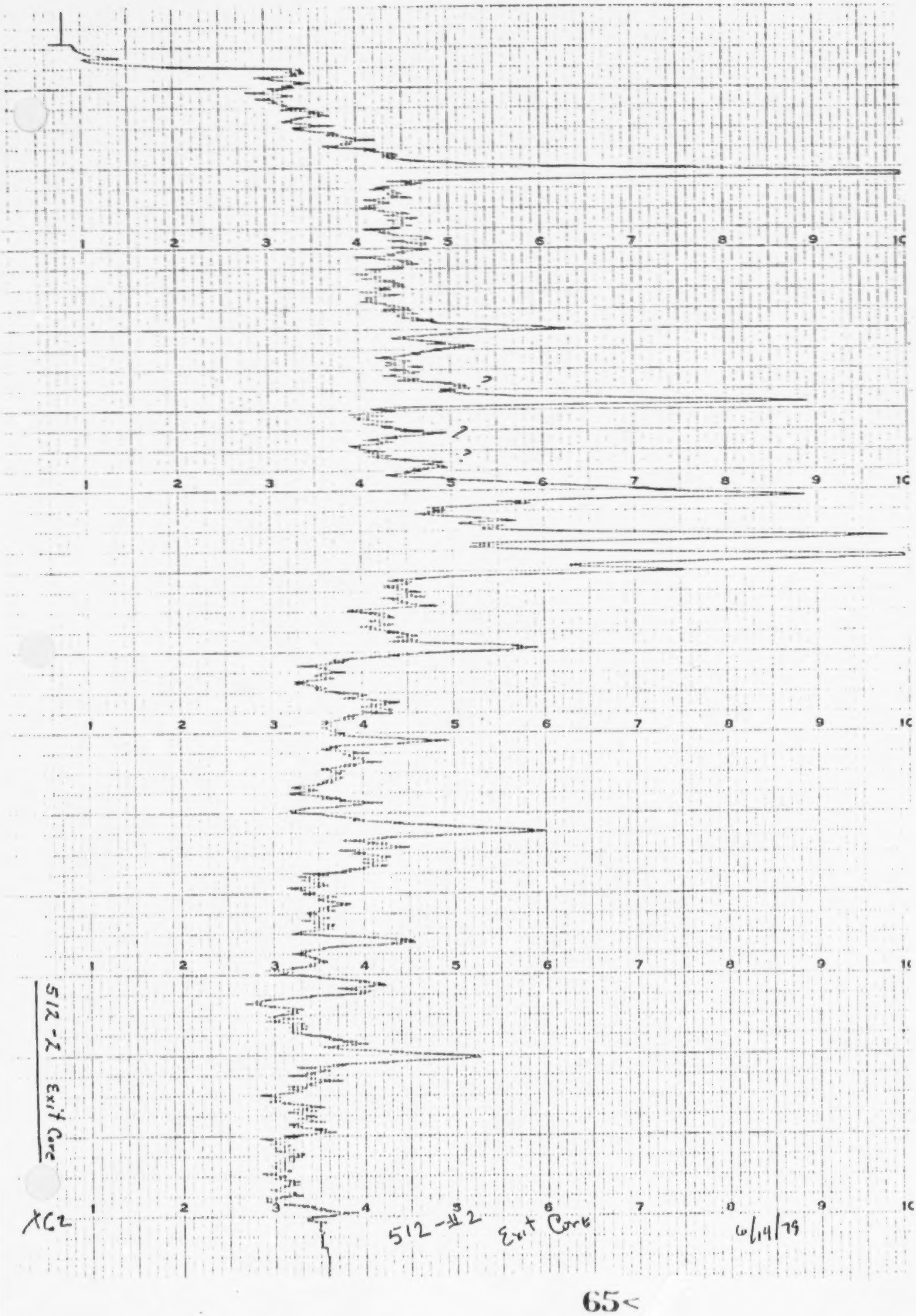
6/11/79



512-1 Inlet deposits

512R1 fuel deposit

6/17/79

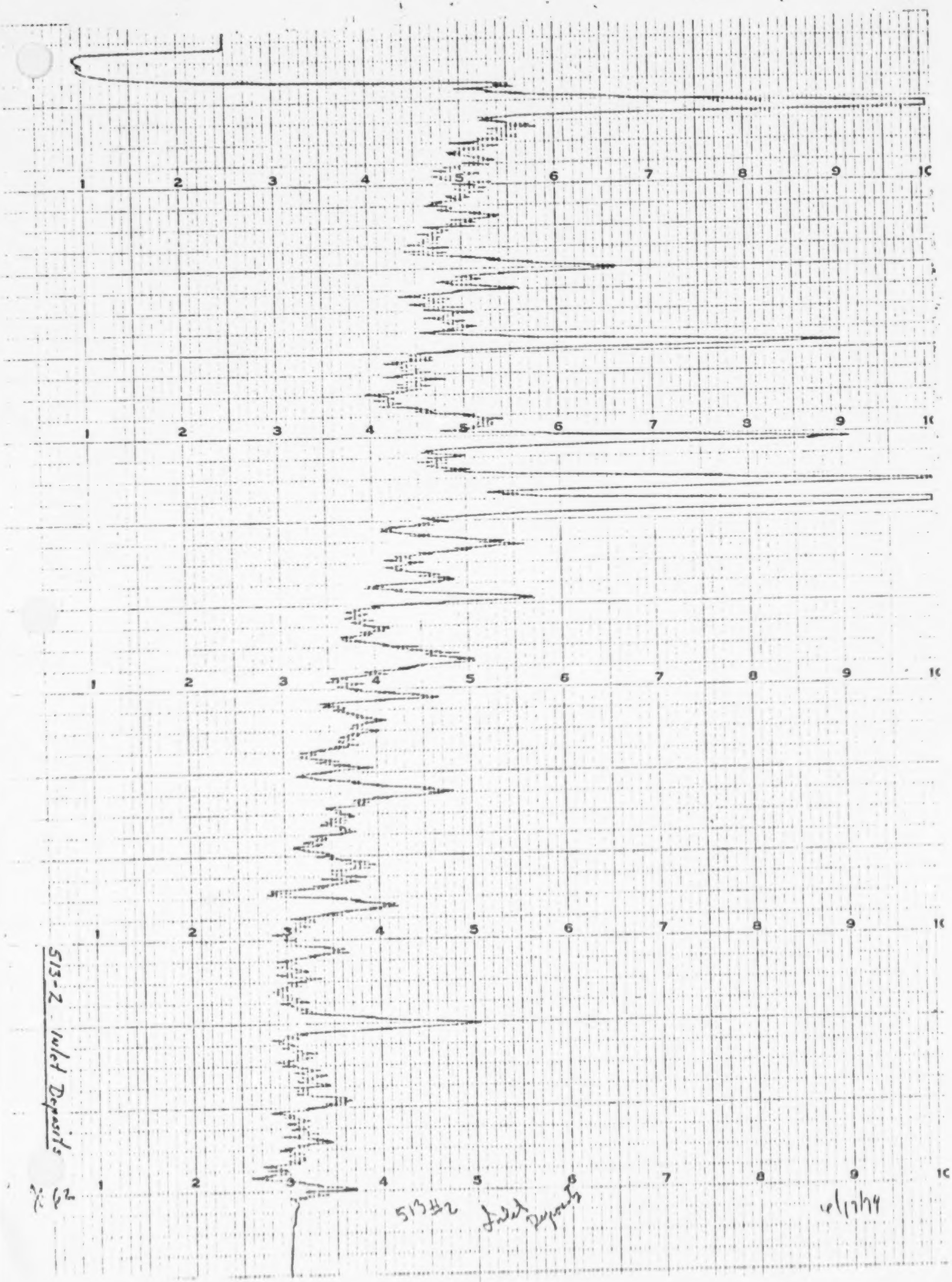


512-2 Exit Core

XC2

512-#2 Exit Core

6/14/79



513-2 Inlet Deposits
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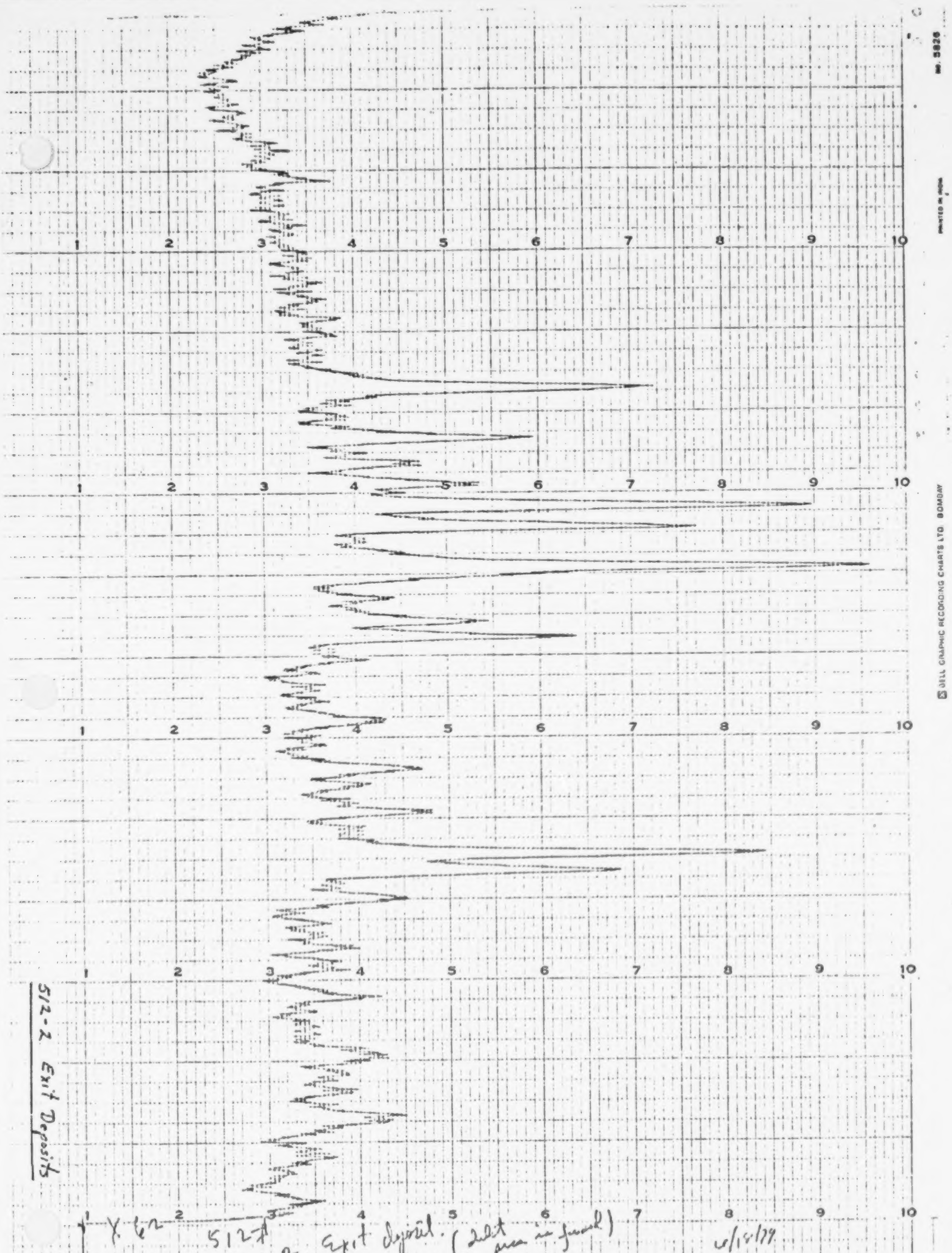
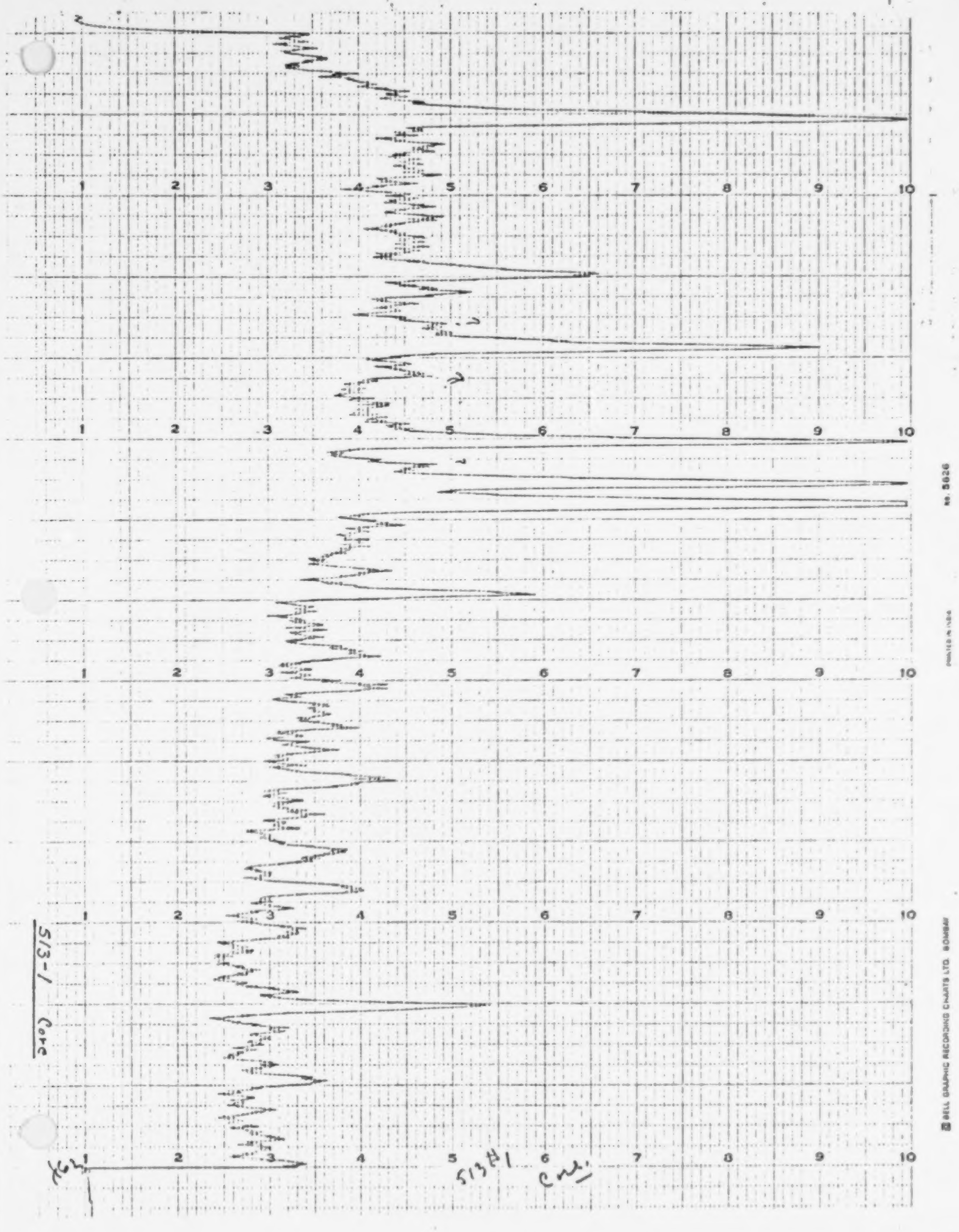
10
513#2 Inlet Deposits
6/17/74



512-2 Exit Deposits

X 62 512-1 2 Exit deposit. (Inlet area in funnel) 6/18/79


1
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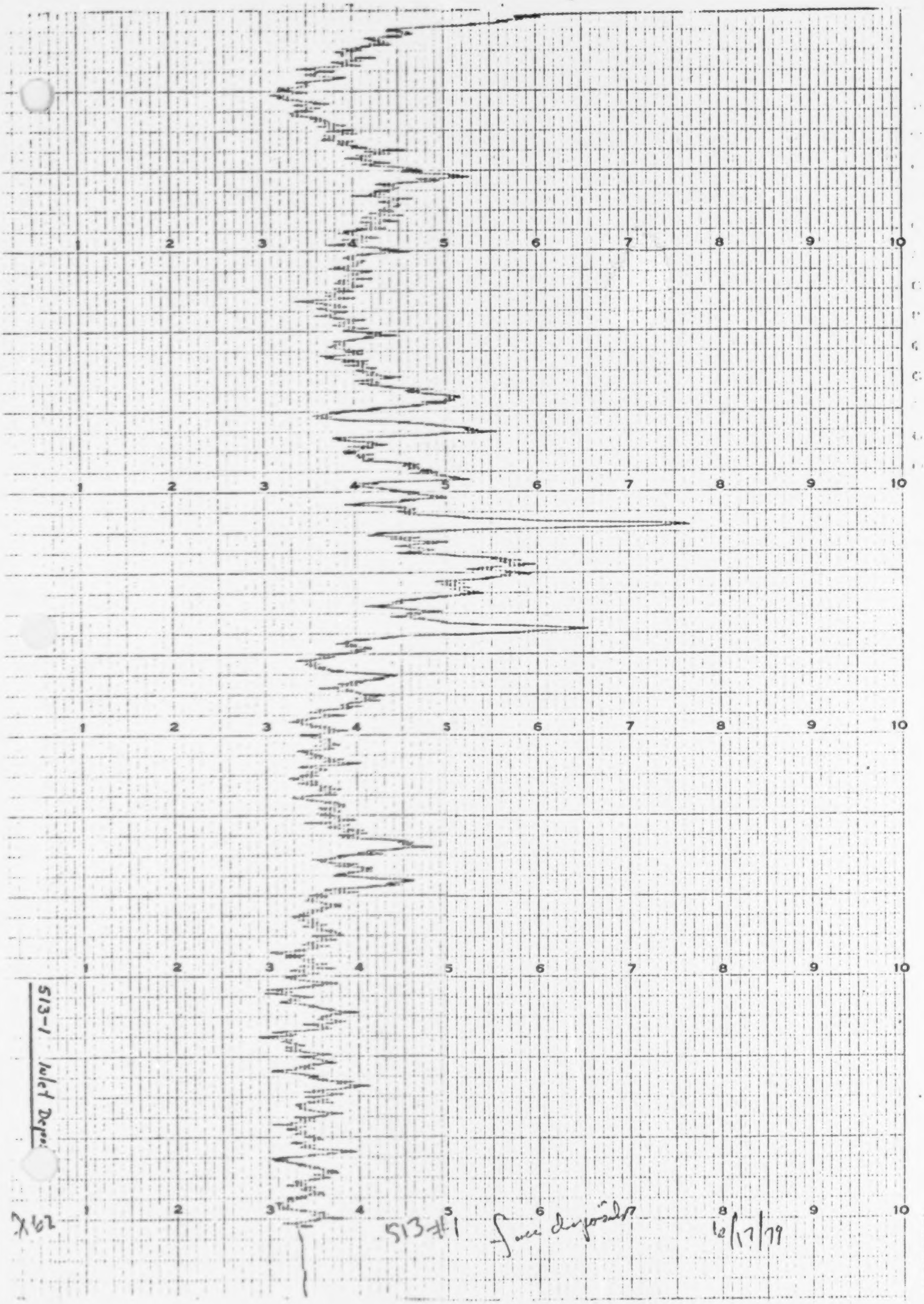
513-1 Core
513#1 Core
No. 5826
PRINTED IN INDIA
BELL GRAPHIC RECORDING CHARTS LTD. BOMBAY

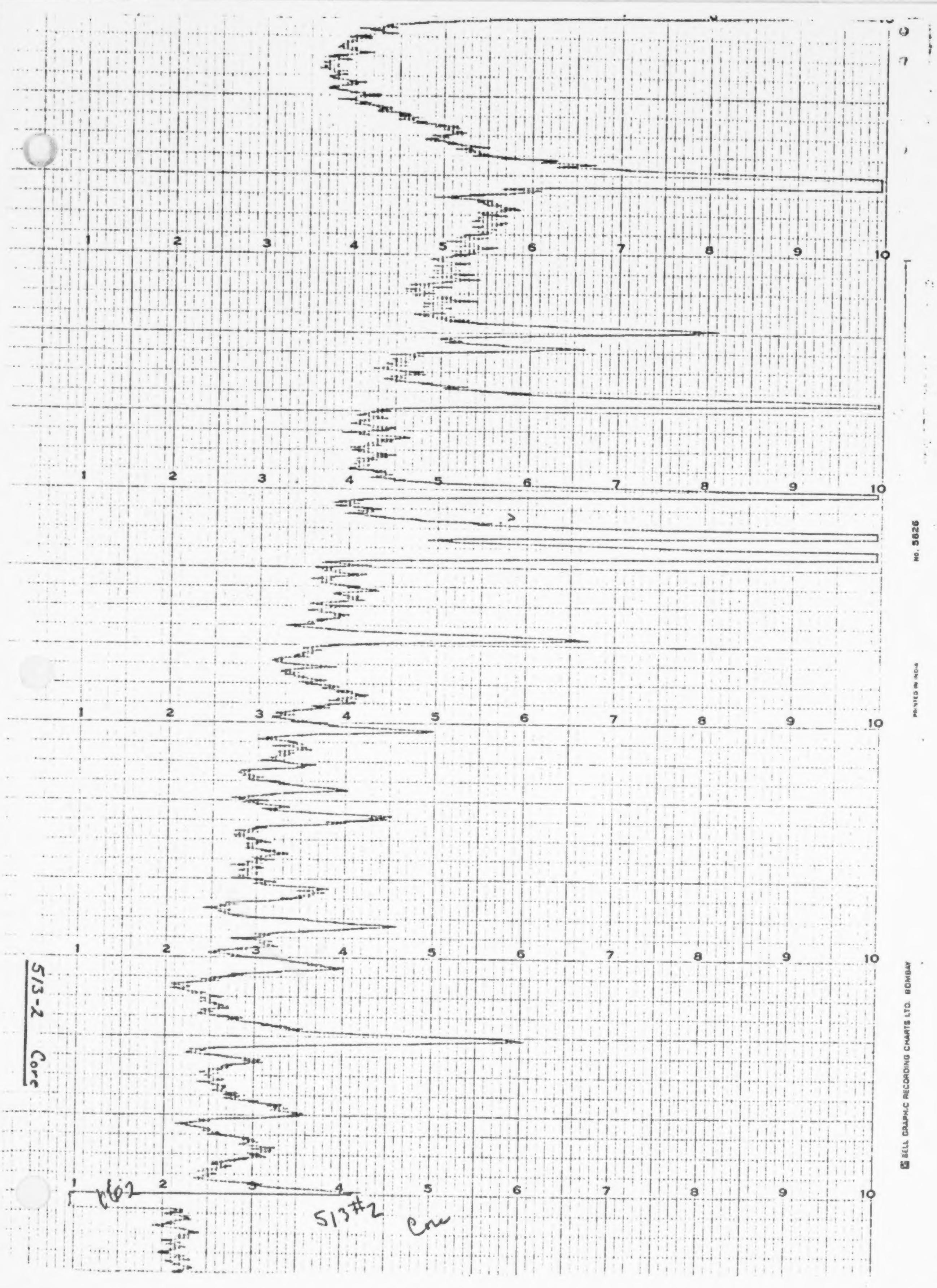


513-1 Inlet Depos

X½

513 #1 fuel deposits

6/17/79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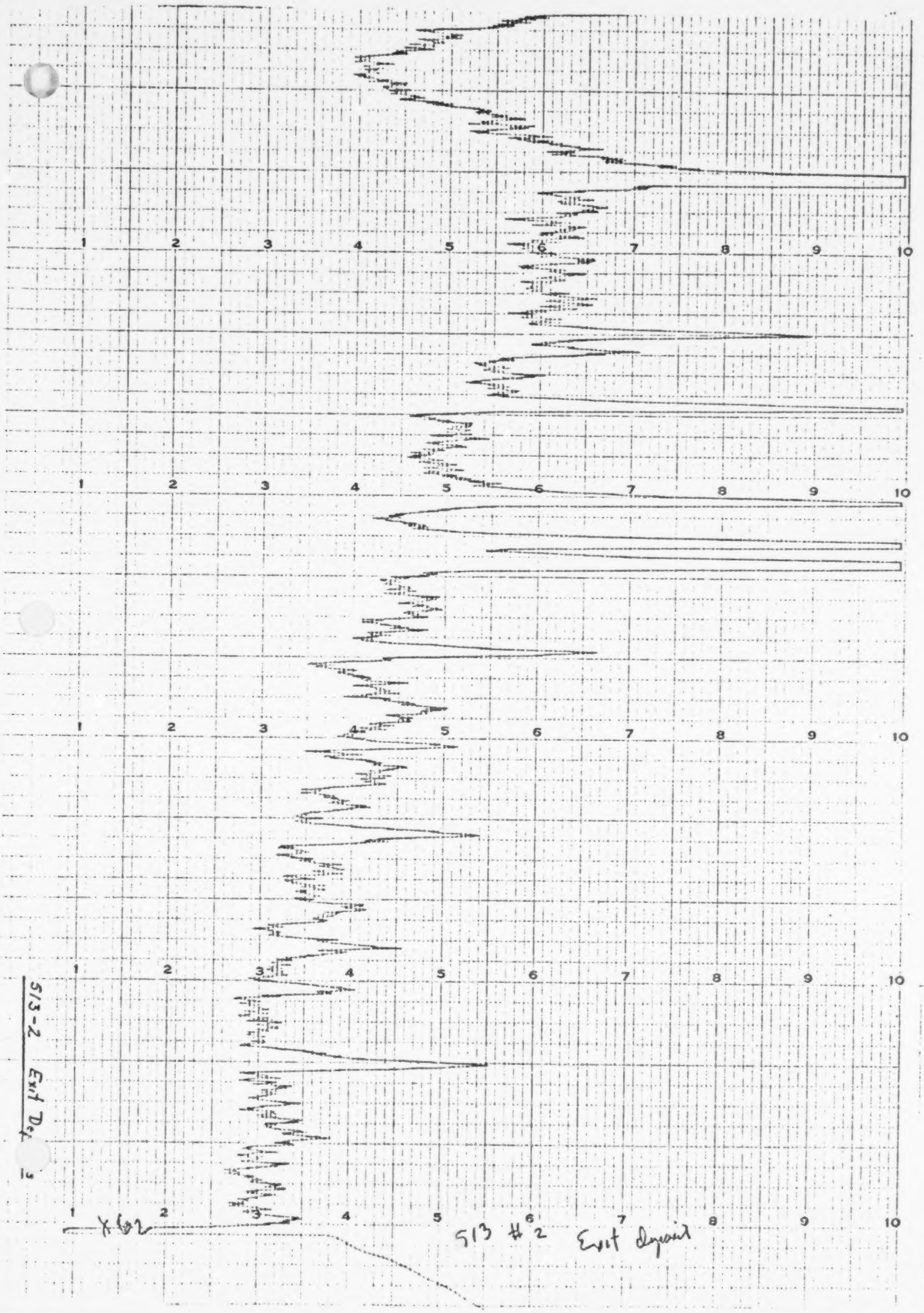
513-2 Core
513#2 Core
NO. 5826
PRINTED IN INDIA
BELL GRAPHIC RECORDING CHARTS LTD. BOMBAY

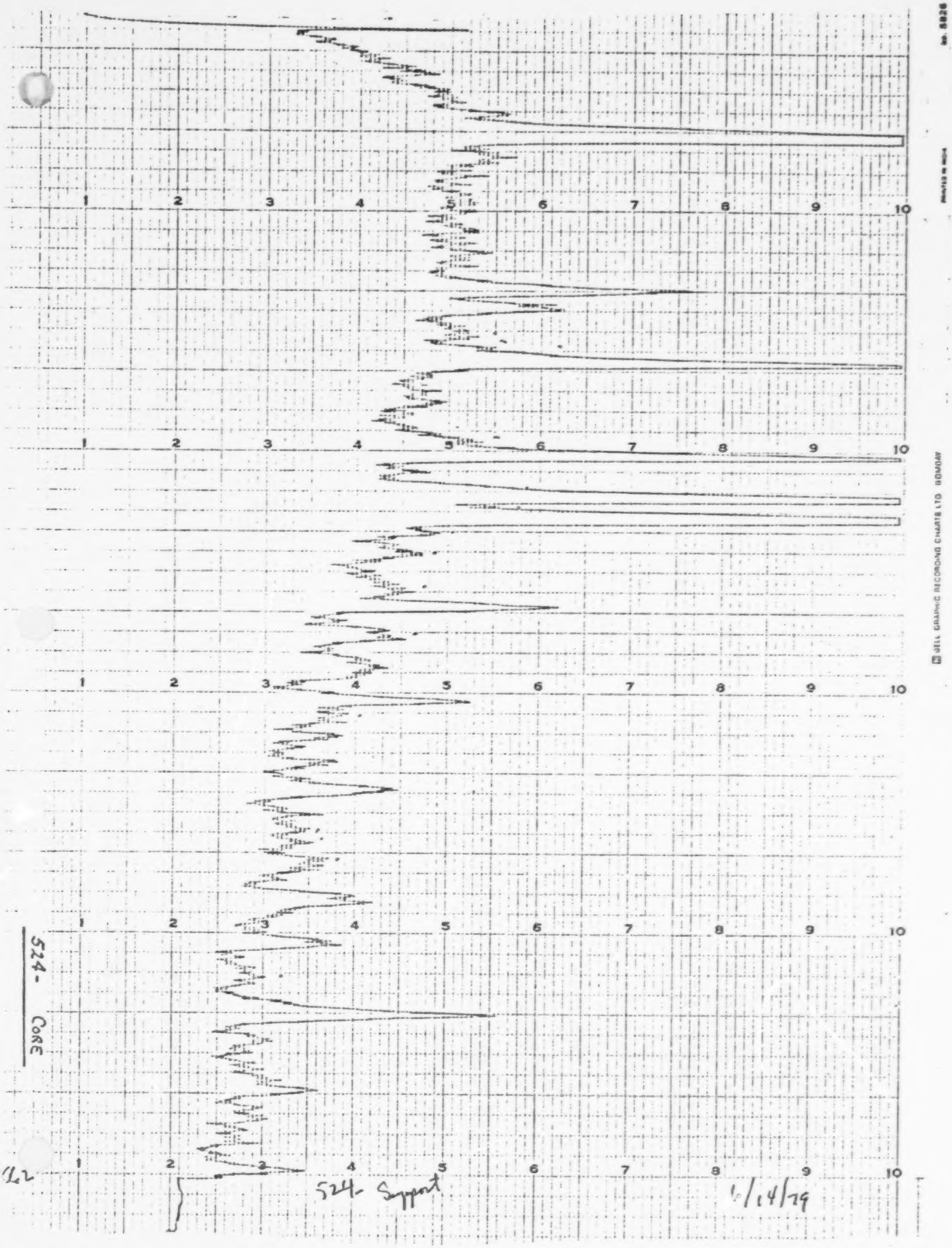


513-2 Exit Dep

X 62

513 #2 Exit deposit

BELL GRAPHIC RECORDING CHARTS LTD. BOMBAY
No. 5826

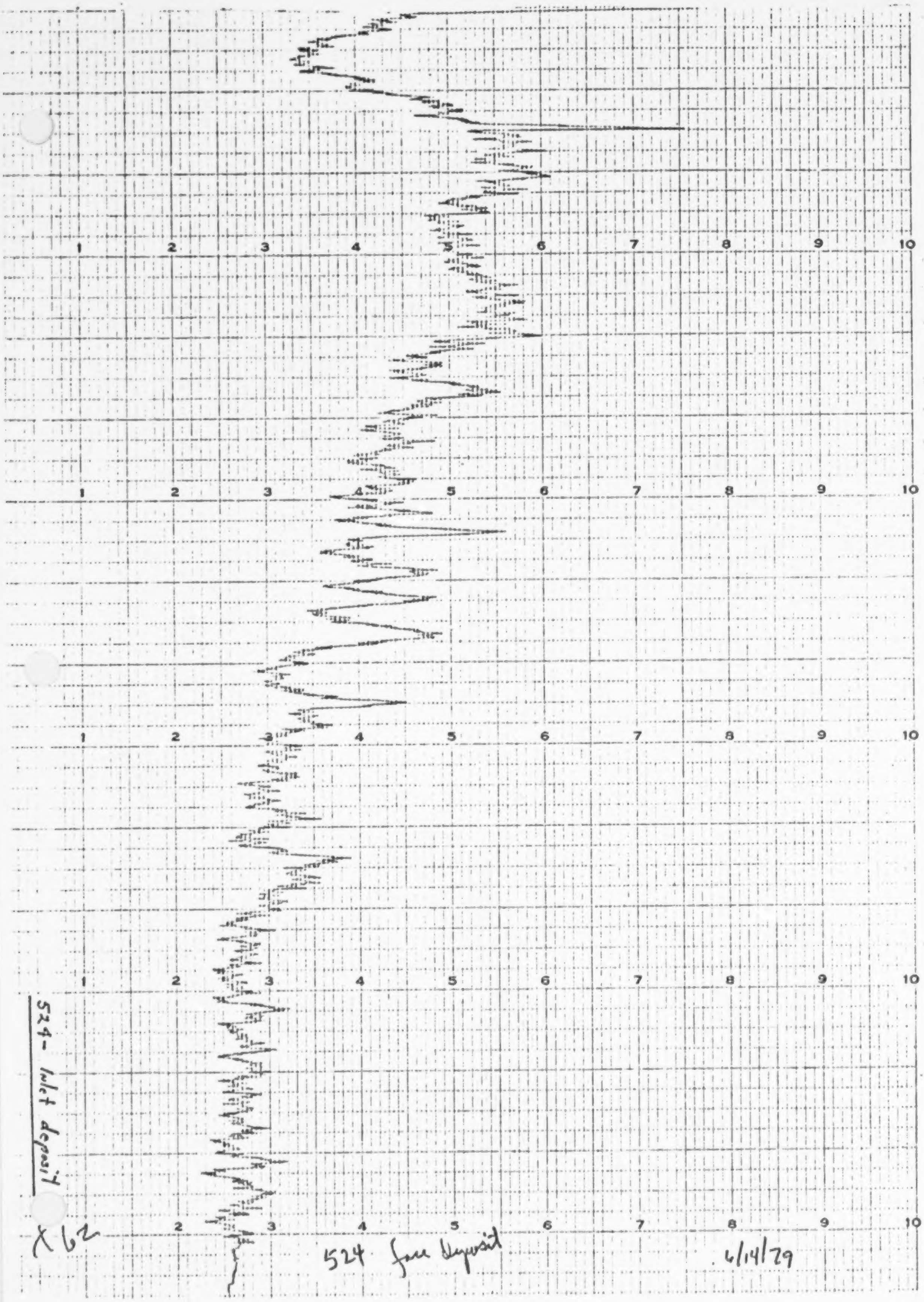


524- Core

1,2

524- Support

6/14/79



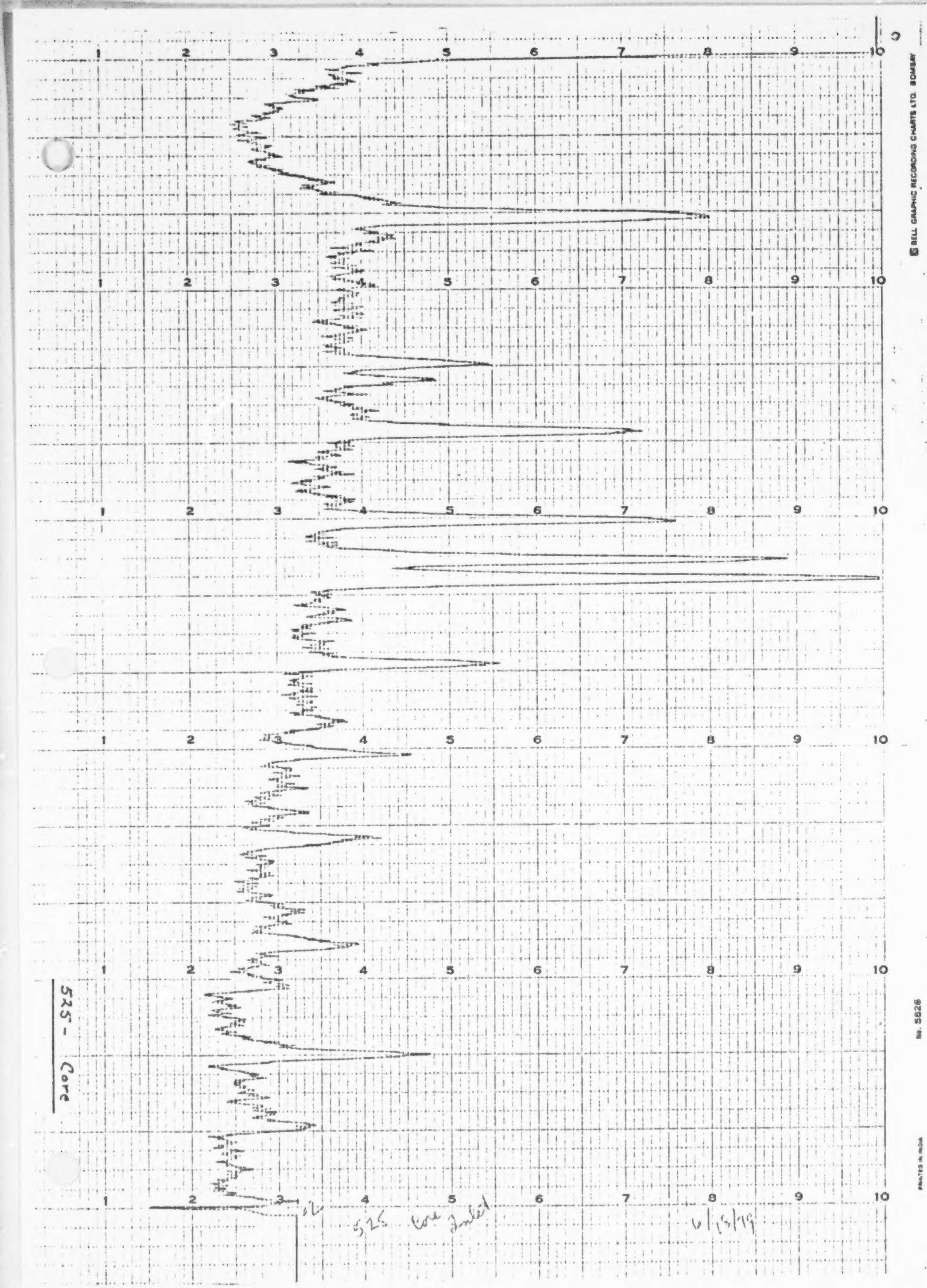
524- Inlet deposit

X 62

524 face deposit

6/14/79

BELL GRAPHIC RECORDING CHARTS LTD. BOMBAY
No. 5826
PRINTED IN INDIA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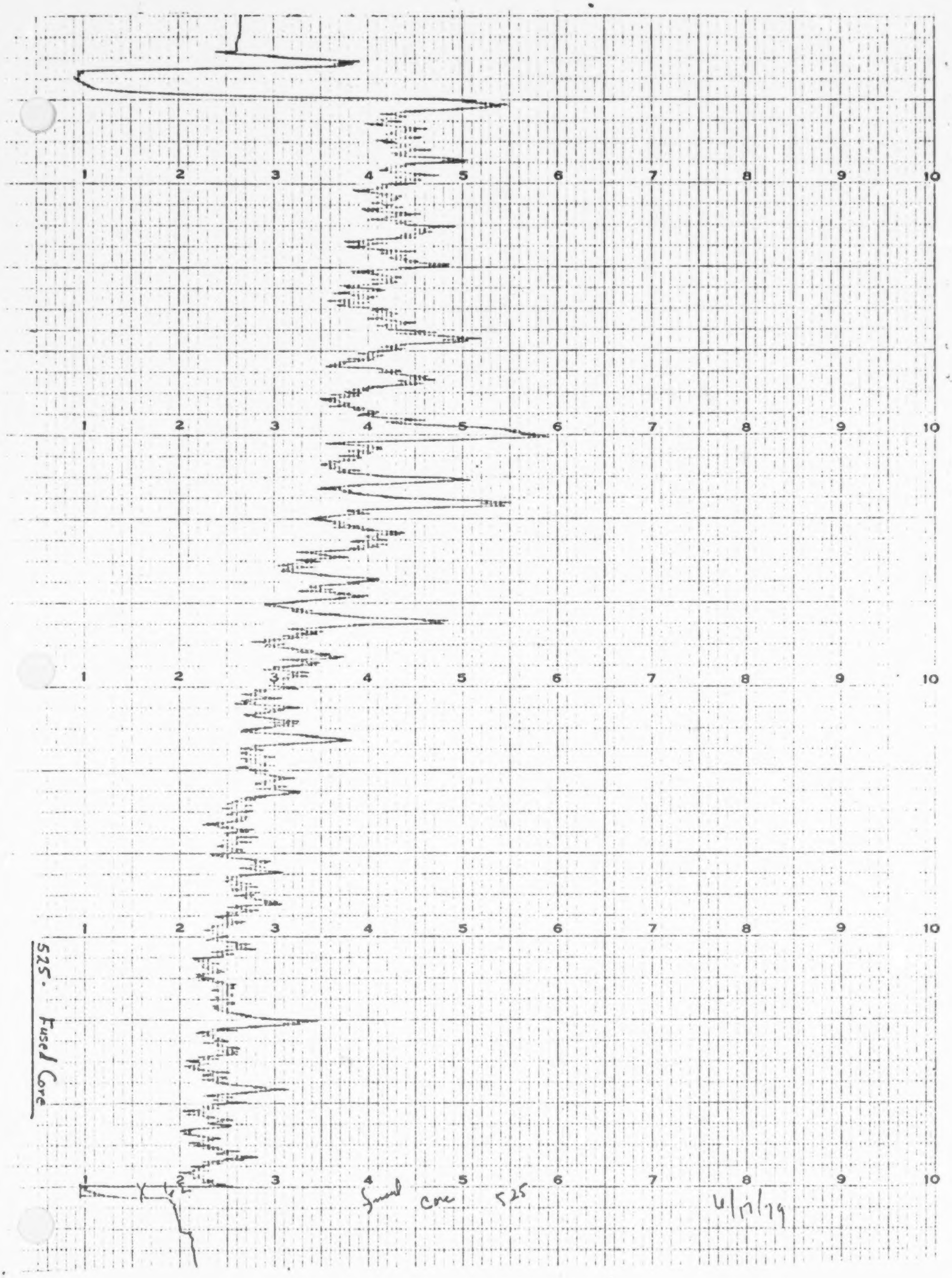
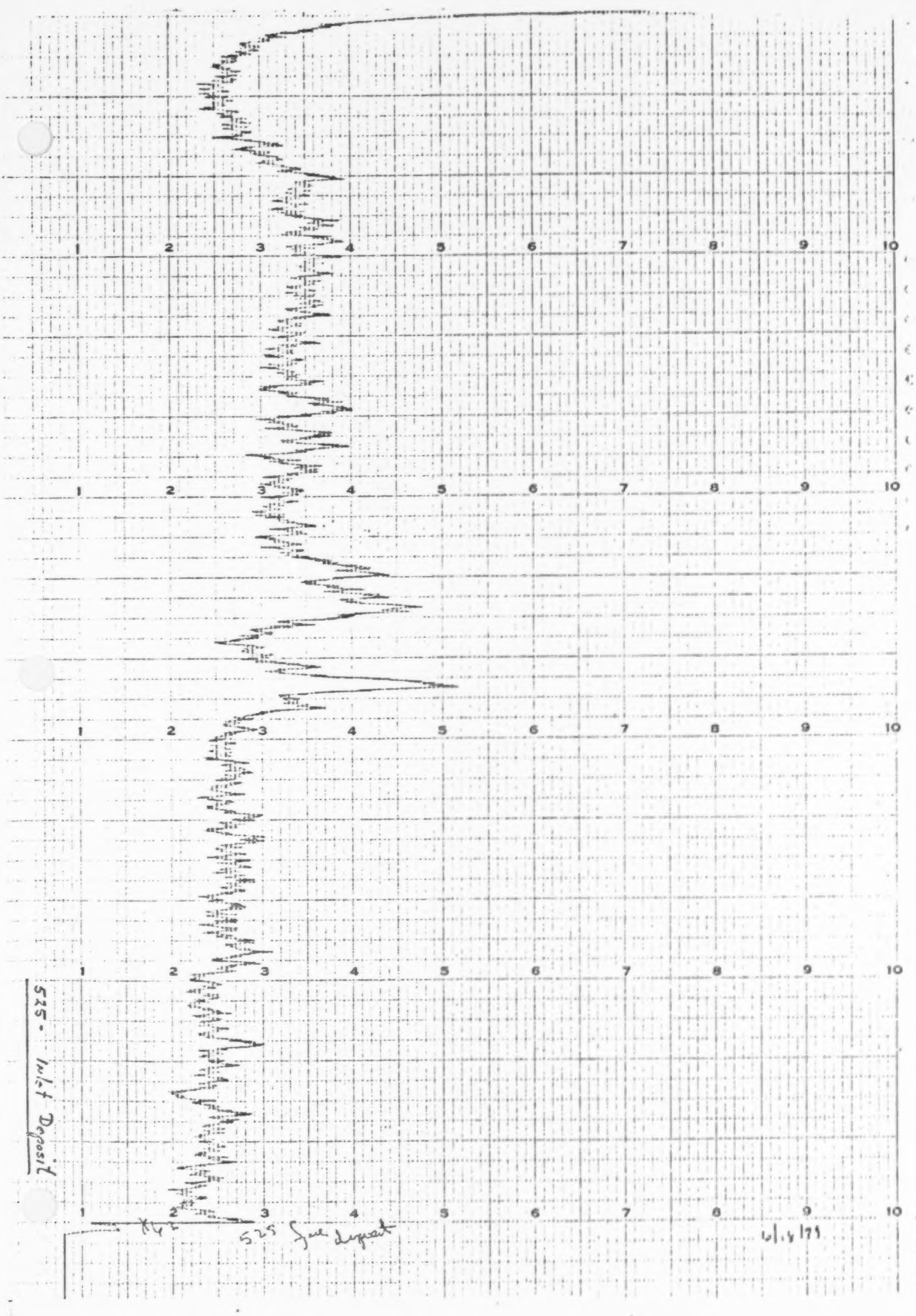
BELL GRAPHIC RECORDING CHARTS LTD. BOMBAY
No. 5826
PRINTED IN INDIA
525- Core
525 Core Inlet
6/13/79



525. Fused Core

fused core 525

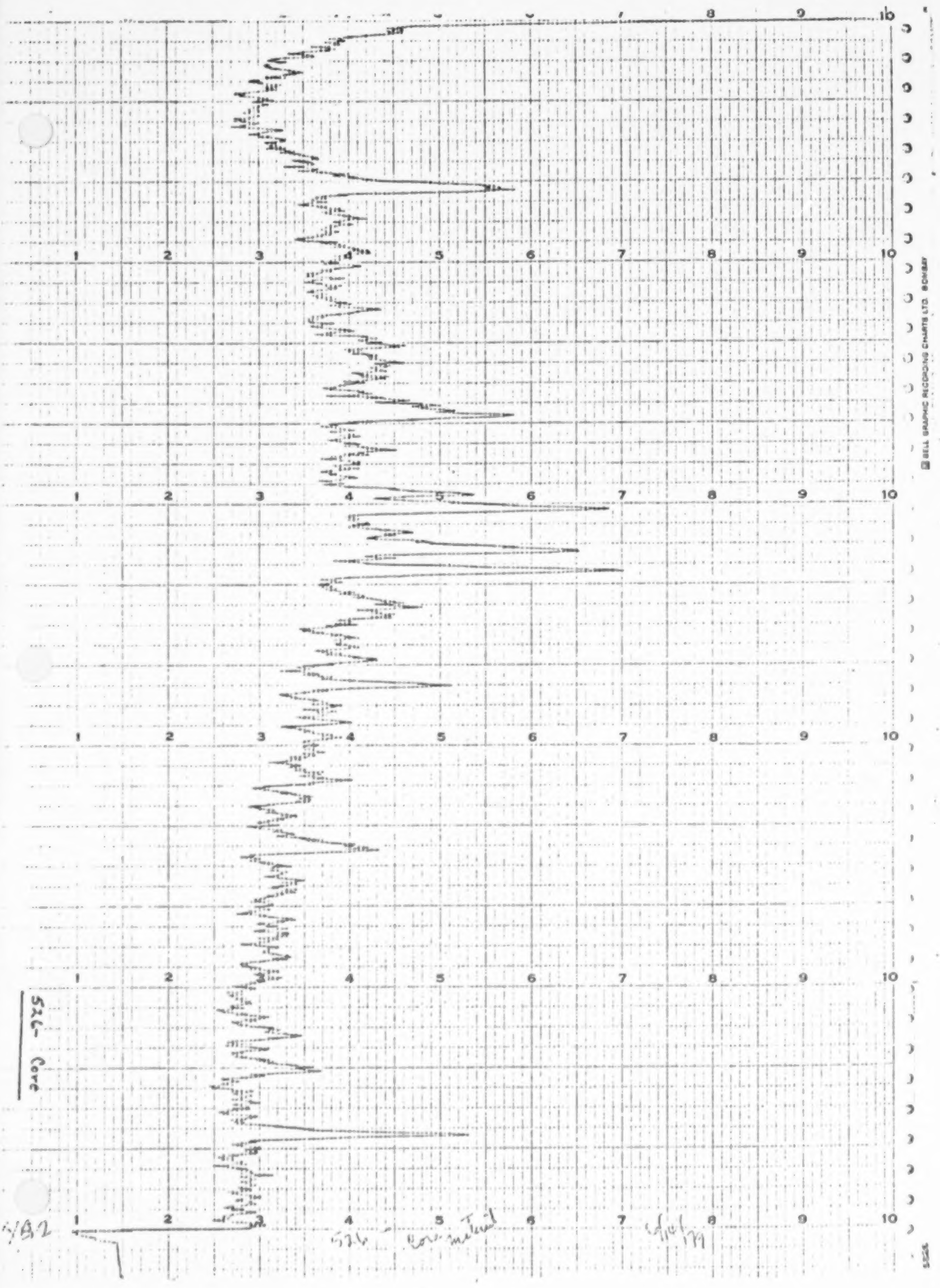
6/17/79



525 - Inlet Deposit

525 fuel deposit

6/8/79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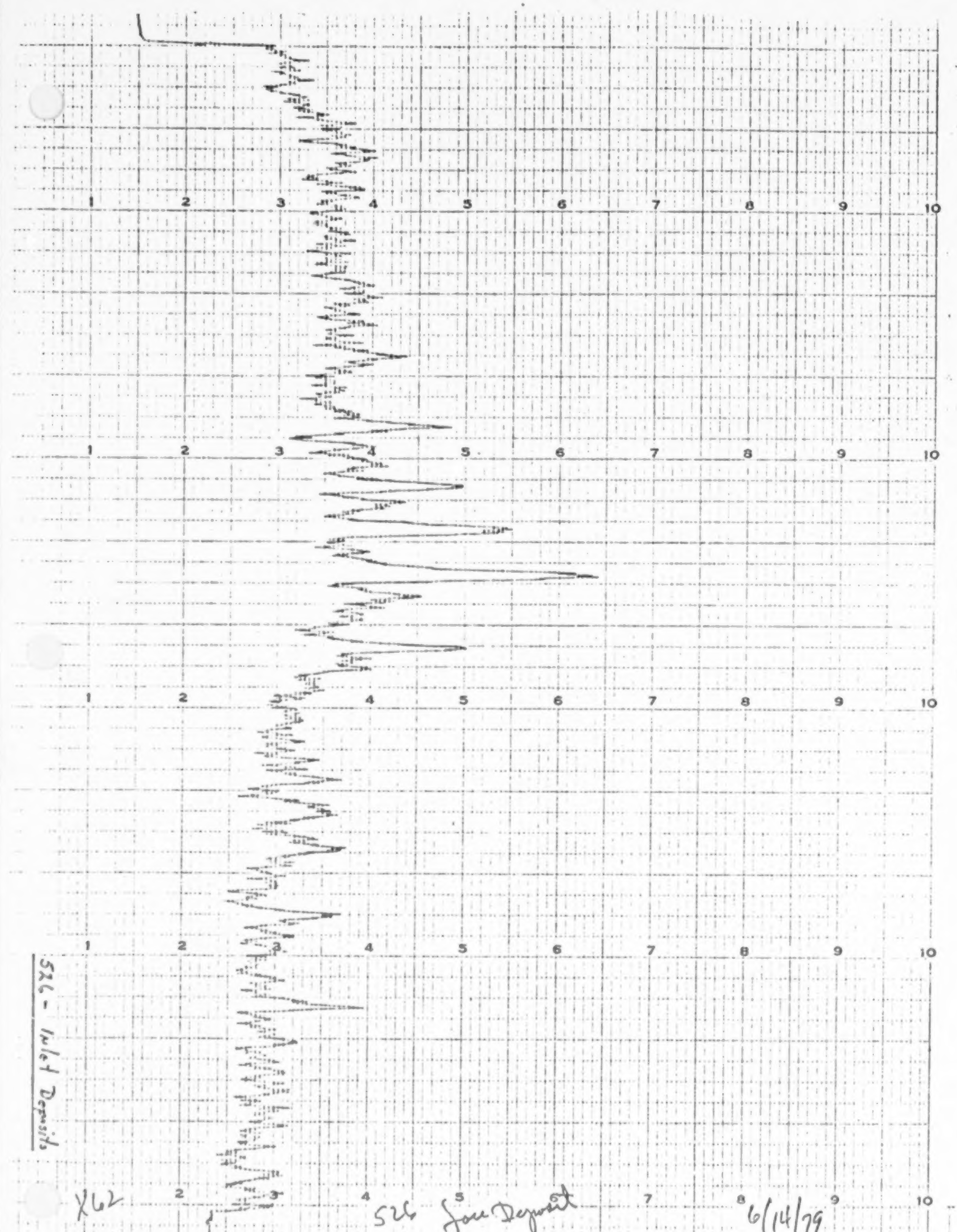
BELL GRAPHIC RECORDING CHARTS LTD. BOMBAY
526- Core
382
526 - core material
5/10/79

77<

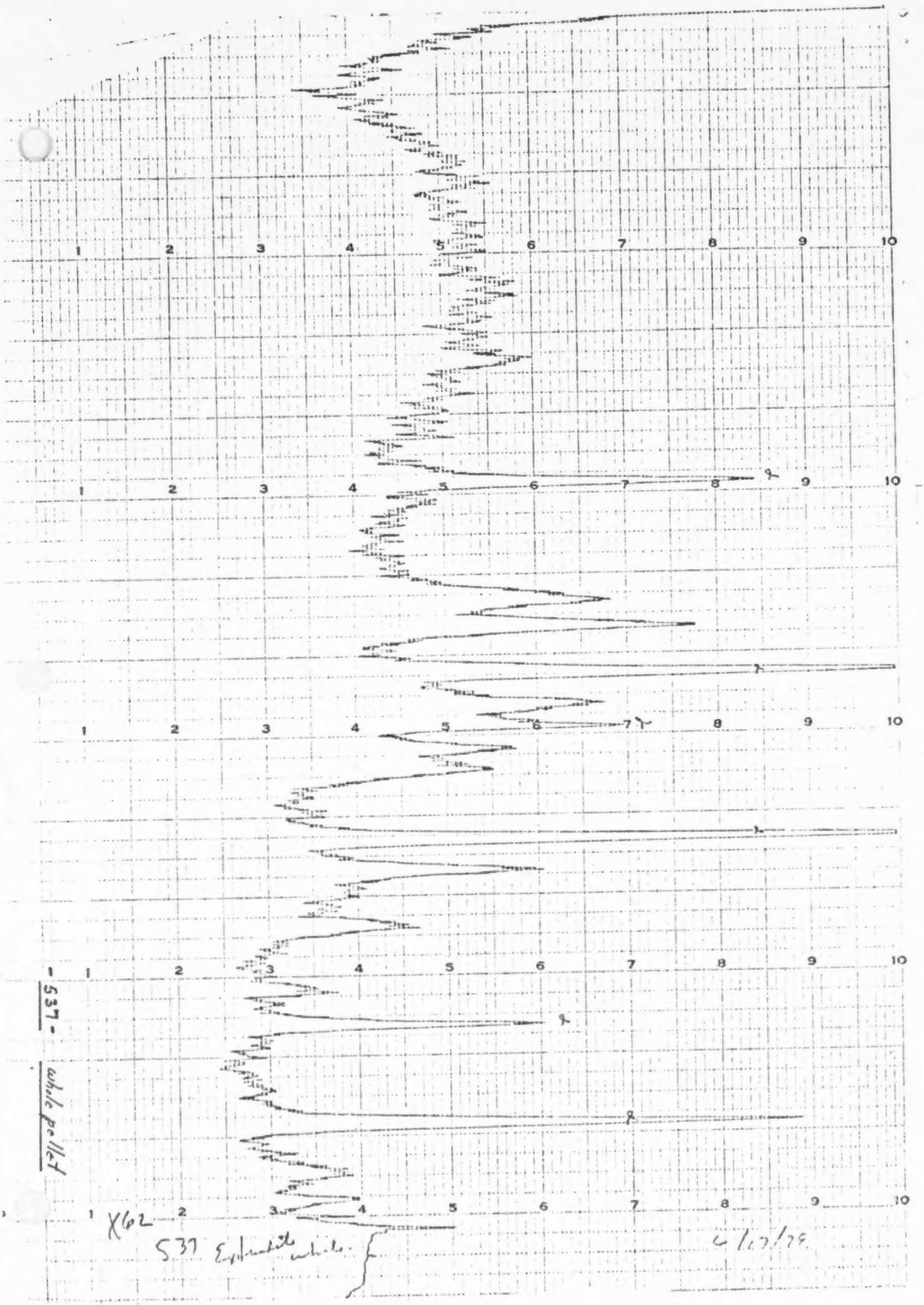


526 - Inlet Deposits

X62

526 Jar Deposit

6/14/79

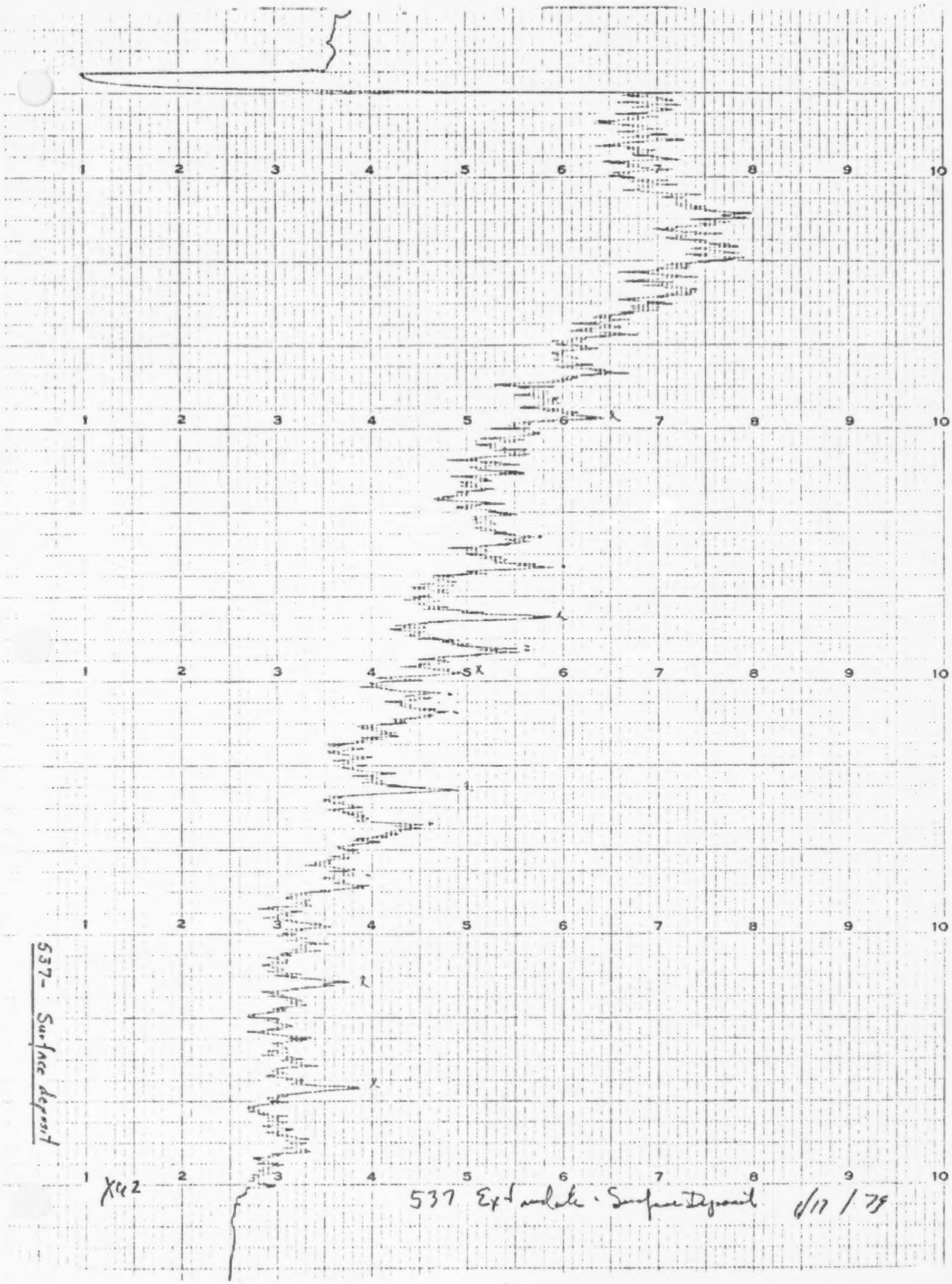


-537- whole pellet

X62

537 Exfoliated whole

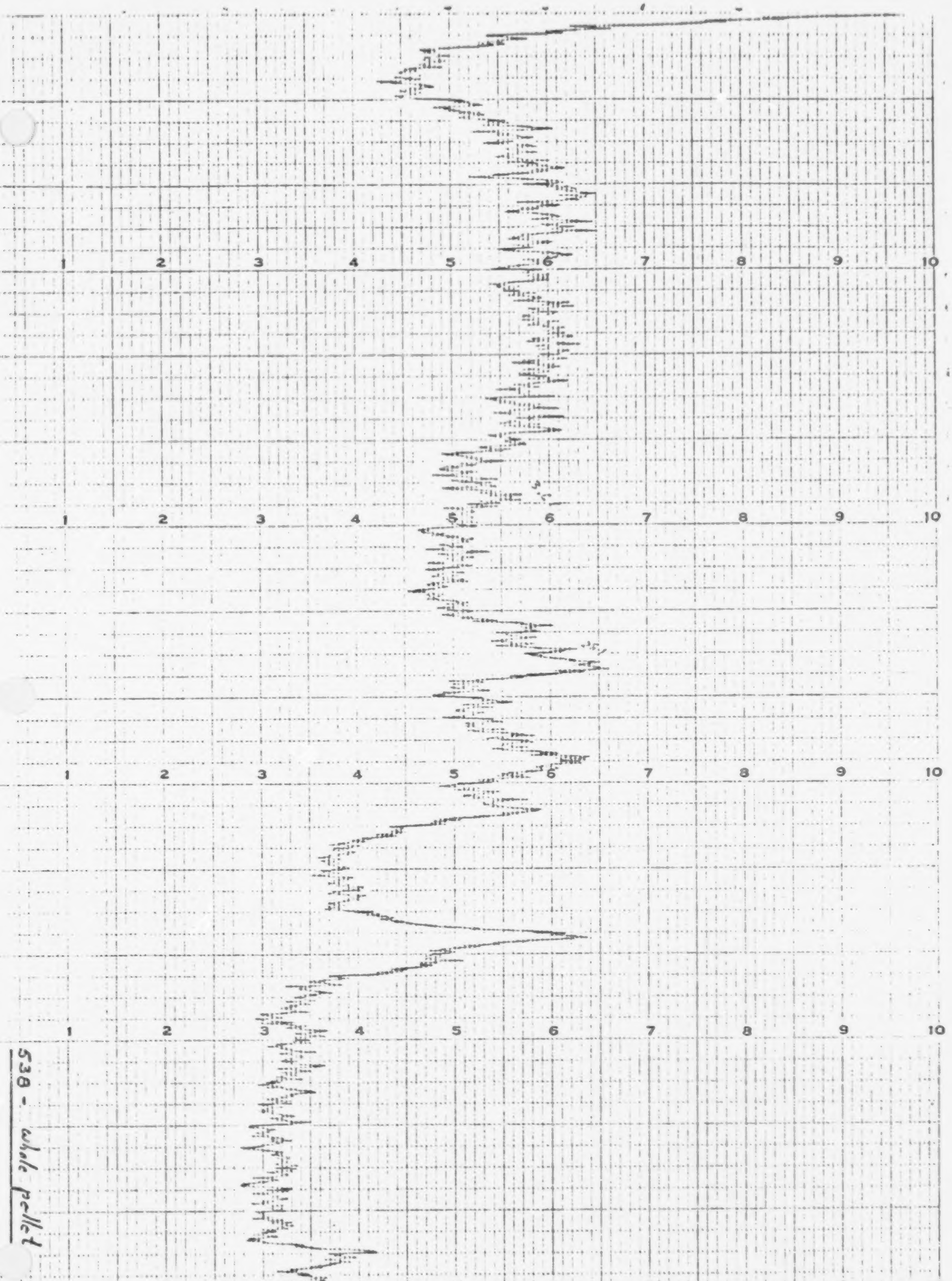
6/17/79



537- Surface deposit

X42

537 Extrudate · Surface Deposit 6/17/79

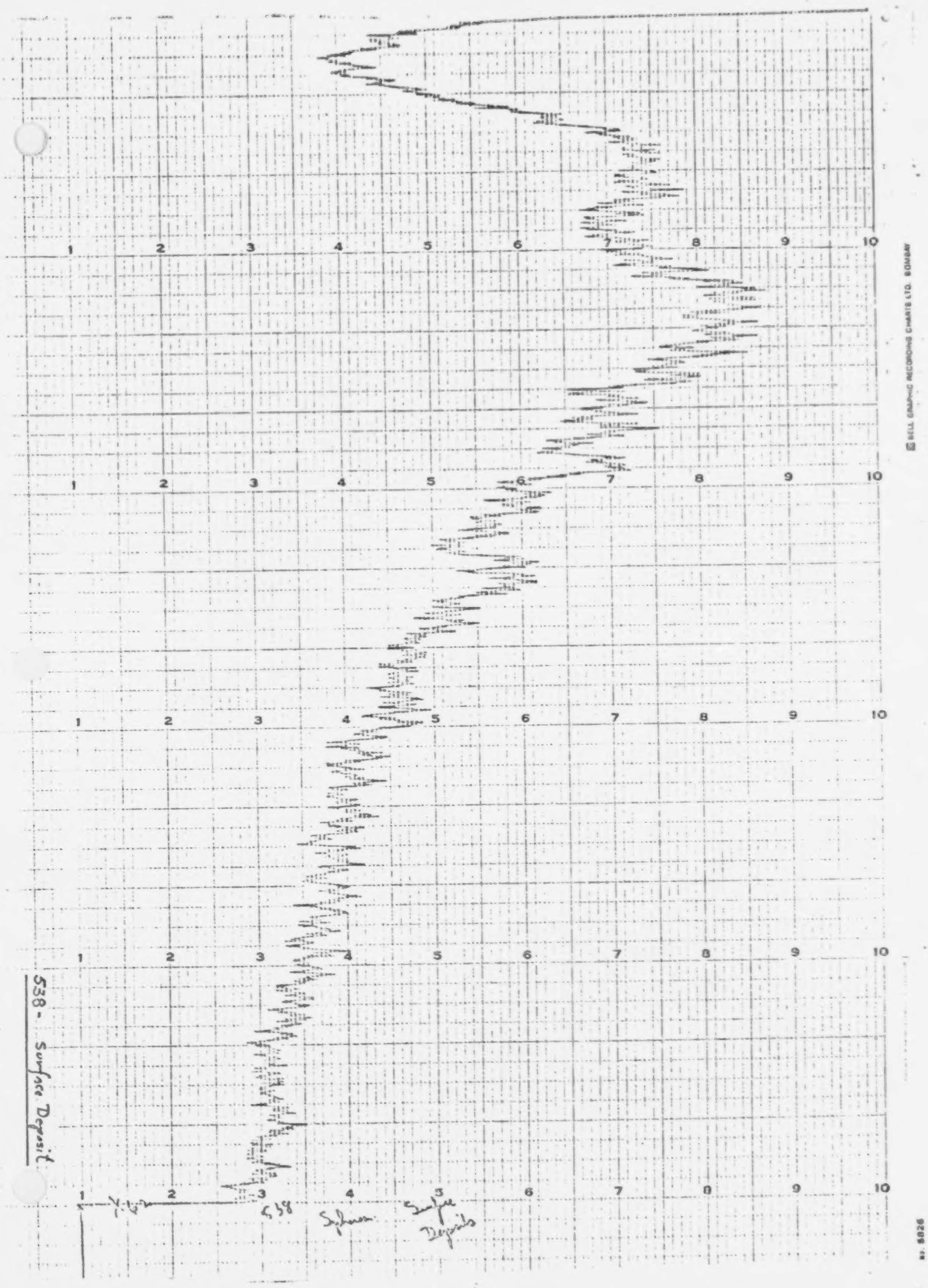


538 - whole pellet

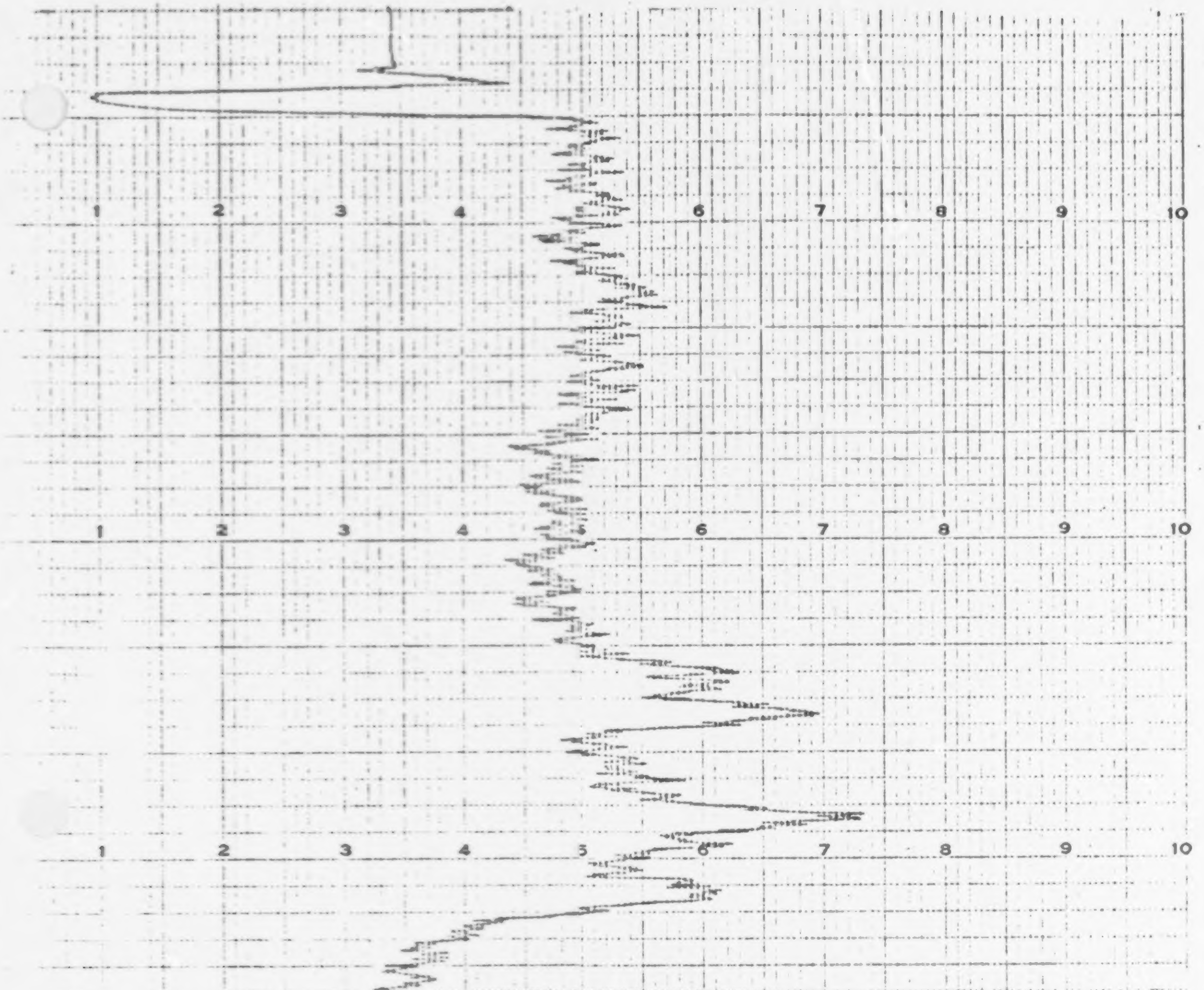
+62 27

538 pellets

1/17/79



538- Surface Deposit



539 whole pellet

Y62

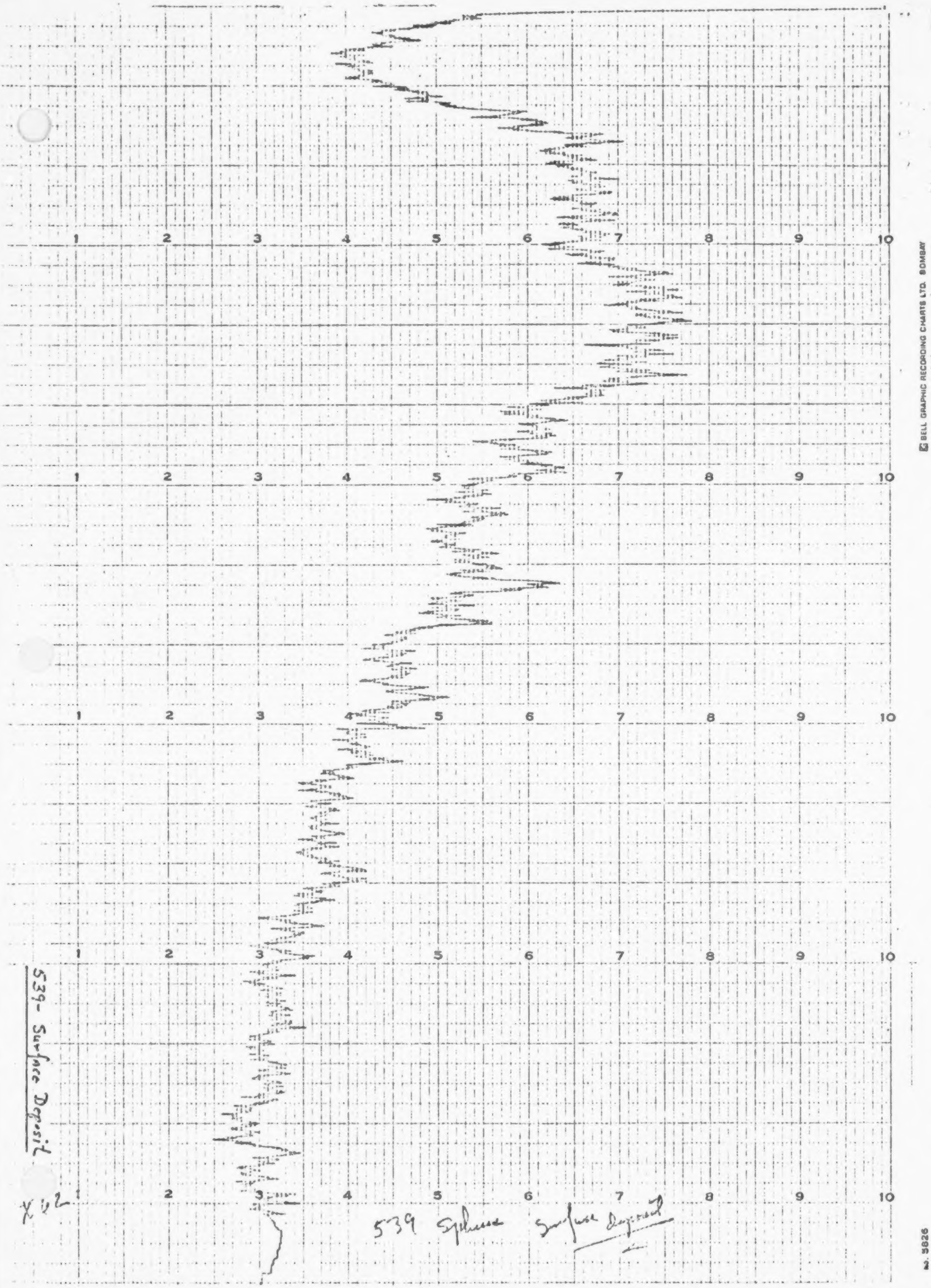
539 Spheres,

6/17/79

NO. 5826

PRINTED IN INDIA

BELL GRAPHIC RECORDING CHARTS LTD. BOMBAY



539- Surface Deposit

X ½

539 Sphene Surface deposit

F. SPECIFIC SURFACE AREA DATA

```
********************** M I C R O M E R I T I C S **********************

       M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                             MODEL 2100D
                  SURFACE AREA DATA AND COMPUTATION

                                                    DATE RUN  07-17-79

 SAMPLE IDENTIFICATION:  SURFACE ANALYSIS & RESEARCH 511-1 PLY

  MANIFOLD VOLUME:  28.050 ML (STP)     EXTRA VOLUME:  131.270 ML (STP)

  ADSORBATE:  NITROGEN

  STATION  2  FLASK NO. #2       OUTGAS TEMP.  220 (C)  OUTGAS TIME   867.  MIN


  W1   21.8364 G        H1  550.0000 MM HG      PS     744.9296 MM HG
                                                                     -1
  W2   20.0000 G        H2  194.3500 MM HG      ALPHA  0.000066 (MM HG)
                                                                 2
  WS    1.8364 G        TS   77.1800 (K)        S        16.2000 A

  VS   11.4302 ML

                   EV      P1        P2         V          X          Y     -1
                        (MM HG)   (MM HG)   (ML, STP)   (P2/PS)    (ML, STP)

  INPUT POINT 1    N    150.2200   20.8180    1.6292   *********  **********

  INPUT POINT 2    N    150.0000   60.4900    1.9244     0.0812    0.045925

  INPUT POINT 3    N    150.0000   88.8900    2.0790     0.1193    0.065172

  INPUT POINT 4    N    155.0000  109.9600    2.1865     0.1476    0.079202

  INPUT POINT 5    N    160.0000  125.8500    2.2696     0.1689    0.089568

  INPUT POINT 6    N    165.0000  138.2800    2.3341     0.1856    0.097656

  SLOPE =      0.4954   INTERCEPT =      0.0059


                         2
  SURFACE AREA =    8.6837 M /G


  STANDARD ERROR OF LEAST SQUARES LINE =  0.000165   % ERROR =  0.2191

  ********** = DATA OUT OF LINEAR REGION OF ISOTHERM,
               NOT USED FOR LEAST SQUARES CALCULATION
```

```
************************ M I C R O M E R I T I C S **********************

        M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                               MODEL 2100D
                    SURFACE AREA DATA AND COMPUTATION

                                                   DATE RUN  07-18-79

 SAMPLE IDENTIFICATION: SURFACE ANALYSIS & RESEARCH 512 DODGE TUBE

  MANIFOLD VOLUME:  28.050 ML (STP)      EXTRA VOLUME: 131.270 ML (STP)

  ADSORBATE  NITROGEN

  STATION  1  FLASK NO. #5      OUTGAS TEMP.  195 (C)  OUTGAS TIME   997.  MIN


  W1    22.6968 G       H1  550.0000 MM HG      PS     746.6702 MM HG
                                                                       -1
  W2    20.0000 G       H2  229.2400 MM HG      ALPHA  0.000066 (MM HG)
                                                                 2
  WS     2.6968 G       TS   77.2000 (K)        S        16.2000 A

  VS     8.3971 ML

                   EV      P1       P2         V          X           Y     -1
                        (MM HG)  (MM HG)  (ML, STP)   (P2/PS)    (ML, STP)

  INPUT POINT 1    N    150.2200  21.6100   1.1955   *********  **********

  INPUT POINT 2    N    150.0000  66.7000   1.4371     0.0893    0.068259

  INPUT POINT 3    N    150.0000  97.1300   1.5572     0.1301    0.096030

  INPUT POINT 4    N    153.0000 117.7500   1.6306     0.1577    0.114822

  INPUT POINT 5    N    156.0000 131.7200   1.6847     0.1764    0.127144


 SLOPE =      0.6773   INTERCEPT =      0.0078

                          2
 SURFACE AREA =    6.3535 M /G

 STANDARD ERROR OF LEAST SQUARES LINE =  0.000137   % ERROR =  0.1352

 ********** = DATA OUT OF LINEAR REGION OF ISOTHERM,
              NOT USED FOR LEAST SQUARES CALCULATION
```

```
*********************** M I C R O M E R I T I C S *********************

     M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                              MODEL 2100D
                  SURFACE AREA DATA AND COMPUTATION

                                                    DATE RUN  07-17-79

 SAMPLE IDENTIFICATION: SURFACE ANALYSIS & RESEARCH 513-1 PLY

  MANIFOLD VOLUME:  28.050 ML (STP)       EXTRA VOLUME: 131.270 ML (STP)

  ADSORBATE: NITROGEN

  STATION  3  FLASK NO.#3       OUTGAS TEMP. 222 (C)  OUTGAS TIME   867. MIN


  W1    22.1061 G        H1  550.0000 MM HG      PS      744.9296 MM HG
                                                                         -1
  W2    20.0000 G        H2  196.7900 MM HG      ALPHA  0.000066 (MM HG)
                                                                    2
  WS     2.1061 G        TS    77.1800 (K)        S        16.2000 A

  VS    11.1829 ML

                       EV       P1        P2        V         X           Y   -1
                             (MM HG)   (MM HG)  (ML,STP)   (P2/PS)     (ML,STP)

  INPUT POINT 1        N    150.2200   22.9340   1.3397   *********  **********
  INPUT POINT 2        N    150.0000   62.9900   1.5689     0.0846    0.058875
  INPUT POINT 3        N    150.0000   91.2000   1.6886     0.1224    0.082618
  INPUT POINT 4        N    155.0000  111.8800   1.7749     0.1502    0.099572
  INPUT POINT 5        N    160.0000  127.5300   1.8368     0.1712    0.112457
  INPUT POINT 6        N    164.0000  139.3900   1.8832     0.1871    0.122233

 SLOPE =      0.6171   INTERCEPT =      0.0068


                                 2
 SURFACE AREA =    6.9765 M /G


 STANDARD ERROR OF LEAST SQUARES LINE =  0.000128   % ERROR =  0.1349

 ********** = DATA OUT OF LINEAR REGION OF ISOTHERM,
              NOT USED FOR LEAST SQUARES CALCULATION
```

```
************************* M I C R O M E R I T I C S ***********************

        M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                                 MODEL 2100D
                       SURFACE AREA DATA AND COMPUTATION

                                                     DATE RUN  07-18-79

 SAMPLE IDENTIFICATION: SURFACE ANALYSIS & RESEARCH 524 FORD

  MANIFOLD VOLUME:  28.050 ML (STP)      EXTRA VOLUME:  131.270 ML (STP)

  ADSORBATE: NITROGEN

  STATION  2  FLASK NO. #6       OUTGAS TEMP.  208 (C)  OUTGAS TIME   997. MIN


  W1    21.1020 G         H1  550.0000 MM HG       PS      746.6702 MM HG
                                                                        -1
  W2    20.0000 G         H2  238.1100 MM HG       ALPHA  0.000066 (MM HG)
                                                                      2
  WS     1.1020 G         TS    77.2000 (K)        S        16.2000 A

  VS     7.7671 ML

                      EV       P1         P2         V          X           Y     -1
                            (MM HG)    (MM HG)   (ML,STP)    (P2/PS)     (ML,STP)

  INPUT POINT 1       N    150.2200    50.2900    1.0070    *********   *********
  INPUT POINT 2       N    150.0000    91.2400    1.1461      0.1222     0.121458
  INPUT POINT 3       N    150.0000   115.4200    1.2214      0.1546     0.149700
  INPUT POINT 4       N    152.0000   130.4000    1.2720      0.1746     0.166345
  INPUT POINT 5       N    154.0000   139.9300    1.3135      0.1874     0.175580

 SLOPE =       0.8355   INTERCEPT =       0.0198

                                  2
 SURFACE AREA =      5.0893 M /G

 STANDARD ERROR OF LEAST SQUARES LINE =  0.000667   % ERROR =  0.4352

 ********* = DATA OUT OF LINEAR REGION OF ISOTHERM,
             NOT USED FOR LEAST SQUARES CALCULATION
```

```
************************  M I C R O M E R I T I C S  **********************

        M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                              MODEL 2100D
                   SURFACE AREA DATA AND COMPUTATION

                                                   DATE RUN  07-17-79

 SAMPLE IDENTIFICATION:  SURFACE ANALYSIS & RESEARCH 525 FORD 400

  MANIFOLD VOLUME:  28. 050 ML (STP)      EXTRA VOLUME: 131. 270 ML (STP)

  ADSORBATE:  NITROGEN

  STATION   4  FLASK NO. #4       OUTGAS TEMP.  153 (C)  OUTGAS TIME   867.  MIN


  W1    24. 3407 G       H1   550. 0000 MM HG      PS      744. 9296 MM HG
                                                                       -1
  W2    20. 0000 G       H2   173. 3600 MM HG      ALPHA  0. 000066 (MM HG)
                                                                 2
  WS     4. 3407 G       TS    77. 1800 (K)        S        16. 2000 A

  VS    13. 2181 ML

                     EV       P1        P2        V          X            Y     -1
                           (MM HG)   (MM HG)  (ML, STP)   (P2/PS)      (ML, STP)

  INPUT POINT 1      N     150. 2200  17. 8980  0. 7174  *********   **********

  INPUT POINT 2      N     150. 0000  54. 6200  0. 8572     0. 0733    0. 092309

  INPUT POINT 3      N     150. 0000  82. 2700  0. 9298     0. 1104    0. 133518

  INPUT POINT 4      N     155. 0000 103. 4700  0. 9816     0. 1389    0. 164332

  INPUT POINT 5      N     160. 0000 120. 0100  1. 0197     0. 1611    0. 188323

  INPUT POINT 6      N     165. 0000 133. 2100  1. 0489     0. 1788    0. 207613

 SLOPE =       1. 0915   INTERCEPT =      0. 0126


                            2
 SURFACE AREA =     3. 9425 M /G


 STANDARD ERROR OF LEAST SQUARES LINE =  0. 000245   % ERROR =  0. 1557

 ********** = DATA OUT OF LINEAR REGION OF ISOTHERM,
              NOT USED FOR LEAST SQUARES CALCULATION
```

```
************************  M I C R O M E R I T I C S  **********************

       M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                              MODEL 2100D
                   SURFACE AREA DATA AND COMPUTATION

                                                     DATE RUN  07-19-79

 SAMPLE IDENTIFICATION: SURFACE ANALYSIS & RESEARCH 526 FORD LTD

  MANIFOLD VOLUME:  28.050 ML (STP)       EXTRA VOLUME: 131.270 ML (STP)

  ADSORBATE: NITROGEN

  STATION  3  FLASK NO.#1       OUTGAS TEMP.  221 (C)  OUTGAS TIME   764.  MIN


  W1    22.3515 G         H1   550.0000 MM HG      PS       746.6702 MM HG
                                                                    -1
  W2    20.0000 G         H2   156.3700 MM HG      ALPHA   0.000066 (MM HG)
                                                                2
  WS     2.3515 G         TS    77.2000 (K)        S        16.2000 A

  VS    16.2784 ML

                     EV       P1         P2          V           X            Y     -1
                           (MM HG)    (MM HG)    (ML,STP)    (P2/PS)      (ML,STP)

  INPUT POINT 1      N     150.2200   13.5630     1.4289    *********   **********

  INPUT POINT 2      N     150.0000   46.3600     1.7181      0.0621     0.038530

  INPUT POINT 3      N     155.0000   74.0100     1.8694      0.0991     0.058858

  INPUT POINT 4      N     161.0000   96.2800     1.9817      0.1289     0.074702

  INPUT POINT 5      N     168.0000  114.7600     2.0668      0.1537     0.087870

  INPUT POINT 6      N     176.0000  130.3400     2.1482      0.1746     0.098444

 SLOPE =        0.5333   INTERCEPT =        0.0057

                              2
 SURFACE AREA =     8.0762 M /G

 STANDARD ERROR OF LEAST SQUARES LINE =  0.000277    % ERROR =  0.3870

 ********** = DATA OUT OF LINEAR REGION OF ISOTHERM,
              NOT USED FOR LEAST SQUARES CALCULATION
```

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*********************** M I C R O M E R I T I C S ***********************

        M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                              MODEL 2100D
                  SURFACE AREA DATA AND COMPUTATION

                                                   DATE RUN  07-18-79

 SAMPLE IDENTIFICATION: SURFACE ANALYSIS & RESEARCH #537  MAL #3153

  MANIFOLD VOLUME:  28.050 ML (STP)       EXTRA VOLUME: 131.270 ML (STP)

  ADSORBATE: NITROGEN

  STATION  4  FLASK NO. D1-D1   OUTGAS TEMP.  188 (C)  OUTGAS TIME   997. MIN


  W1    22.8531 G         H1  550.0000 MM HG      PS      746.6702 MM HG
                                                                        -1
  W2    22.7337 G         H2  141.9900 MM HG      ALPHA  0.000066 (MM HG)
                                                                    2
  WS     0.1194 G         TS    77.2000 (K)       S        16.2000 A

  VS    18.7894 ML

                     EV       P1        P2        V          X           Y    -1
                           (MM HG)   (MM HG)  (ML, STP)   (P2/PS)     (ML, STP)

  INPUT POINT 1      N     150.2200   28.1380  11.2730  *********   **********

  INPUT POINT 2      N     150.0000   57.9900  12.8482     0.0777     0.006554

  INPUT POINT 3      N     150.0000   80.7200  13.7760     0.1081     0.008799

  INPUT POINT 4      N     155.0000   99.0000  14.5637     0.1326     0.010496

  INPUT POINT 5      N     160.0000  114.0300  15.1670     0.1527     0.011884

  INPUT POINT 6      N     165.0000  126.5900  15.6529     0.1695     0.013042

  INPUT POINT 7      N     170.0000  137.3200  16.0192     0.1839     0.014068

 SLOPE =       0.0704    INTERCEPT =      0.0011

                         2
 SURFACE AREA =   60.8874 M /G

 STANDARD ERROR OF LEAST SQUARES LINE =  0.000034    % ERROR =   0.3123

 ********** = DATA OUT OF LINEAR REGION OF ISOTHERM,
              NOT USED FOR LEAST SQUARES CALCULATION
```

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************************  M I C R O M E R I T I C S  ***********************

        M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                              MODEL 2100D
                  SURFACE AREA DATA AND COMPUTATION

                                                      DATE RUN  07-18-79

 SAMPLE IDENTIFICATION: SURFACE ANALYSIS & RESEARCH #538   MAL #3150

  MANIFOLD VOLUME:  28.050 ML (STP)       EXTRA VOLUME: 131.270 ML (STP)

  ADSORBATE: NITROGEN

  STATION  3  FLASK NO. L-L     OUTGAS TEMP. 212 (C)  OUTGAS TIME   997. MIN


  W1   19.1163 G       H1  550.0000 MM HG     PS      746.6702 MM HG
                                                                      -1
  W2   19.0054 G       H2  147.4200 MM HG     ALPHA  0.000066 (MM HG)
                                                                 2
  WS    0.1109 G       TS   77.2000 (K)       S        16.2000 A

  VS   17.7837 ML
                     EV      P1        P2         V          X           Y     -1
                          (MM HG)   (MM HG)   (ML, STP)   (P2/PS)    (ML, STP)

  INPUT POINT 1      N    150.2200   22.0990   20.0059  *********  **********

  INPUT POINT 2      N    150.0000   53.4600   23.0992     0.0716    0.003339

  INPUT POINT 3      N    150.0000   77.5600   24.8937     0.1039    0.004656

  INPUT POINT 4      N    155.0000   96.8600   26.3273     0.1297    0.005662

  INPUT POINT 5      N    160.0000  112.8200   27.2194     0.1511    0.006539

  INPUT POINT 6      N    165.0000  125.9300   28.0266     0.1687    0.007239

  INPUT POINT 7      N    170.0000  136.8200   28.8988     0.1832    0.007763

  SLOPE -      0.0398   INTERCEPT =      0.0005

                           2
  SURFACE AREA -  108.0321 M /G

  STANDARD ERROR OF LEAST SQUARES LINE =  0.000021    % ERROR =  0.3583

  ********** = DATA OUT OF LINEAR REGION OF ISOTHERM,
               NOT USED FOR LEAST SQUARES CALCULATION
```

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************************ M I C R O M E R I T I C S **********************

         M A T E R I A L S   A N A L Y S I S   L A B O R A T O R Y

                                 MODEL 2100D
                     SURFACE AREA DATA AND COMPUTATION

                                                      DATE RUN  07-18-79

 SAMPLE IDENTIFICATION:  SURFACE ANALYSIS & RESEARCH #539  #3152

  MANIFOLD VOLUME:   28.050 ML (STP)       EXTRA VOLUME:  131.270 ML (STP)

  ADSORBATE:  NITROGEN

  STATION  4  FLASK NO. 33K-T   OUTGAS TEMP.  190 (C)  OUTGAS TIME   764.  MIN


  W1    18.9970 G          H1   550.0000 MM HG       PS        746.6702 MM HG
                                                                          -1
  W2    18.8689 G          H2   135.6900 MM HG       ALPHA  0.000066 (MM HG)
                                                                      2
  WS     0.1281 G          TS    77.2000 (K)         S         16.2000 A

  VS    20.0571 ML

                     EV      P1        P2         V          X           Y    -1
                          (MM HG)   (MM HG)   (ML,STP)   (P2/PS)     (ML,STP)

  INPUT POINT 1      N    150.2300   26.5980   10.8140   *********  **********

  INPUT POINT 2      N    150.0000   55.4800   12.3101     0.0743    0.006520

  INPUT POINT 3      N    155.0000   78.9600   13.2621     0.1057    0.008917

  INPUT POINT 4      N    160.0000   98.0100   14.0714     0.1313    0.010738

  INPUT POINT 5      N    165.0000  113.8800   14.5854     0.1525    0.012339

  INPUT POINT 6      N    170.0000  127.0900   15.0864     0.1702    0.013597

  INPUT POINT 7      N    175.0000  136.3500   15.5193     0.1853    0.014655

 SLOPE =       0.0733   INTERCEPT =       0.0011

                             2
 SURFACE AREA =    58.5069 M /G

 STANDARD ERROR OF LEAST SQUARES LINE =  0.000036   % ERROR =  0.3262

 ********** = DATA OUT OF LINEAR REGION OF ISOTHERM,
              NOT USED FOR LEAST SQUARES CALCULATION
```

G. DIFFERENTIAL THERMAL ANALYSIS CURVES

PART NO. 990092

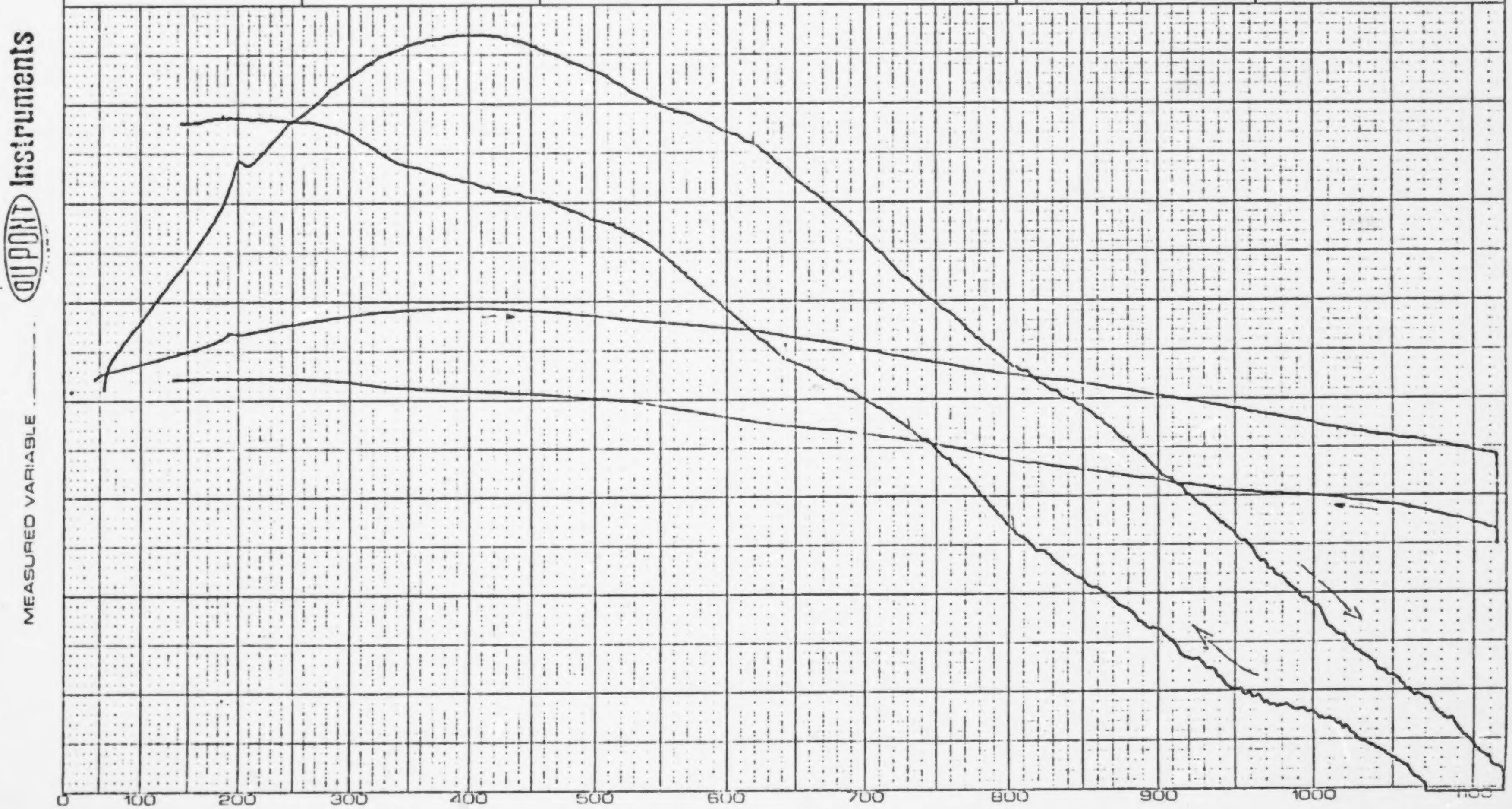
RUN NO. ___ DATE 6/20/75
OPERATOR A. Rullat
SAMPLE: EPA - 511
ATM 0 8% O_2 ___
FLOW RATE ___

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in 140.2
WEIGHT, mg 31.00
REFERENCE Aluminum Oxide

TGA
SCALE, mg/in ___
SUPPRESSION, mg ___
WEIGHT, mg ___
TIME CONST., sec ___
dY, (mg/min)/in ___

TMA
SCALE, mils/in ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___



DU PONT Instruments

PART NO. 990092

RUN NO.___ DATE 6/20/79
OPERATOR A. Lalley
SAMPLE: EPA 512 I

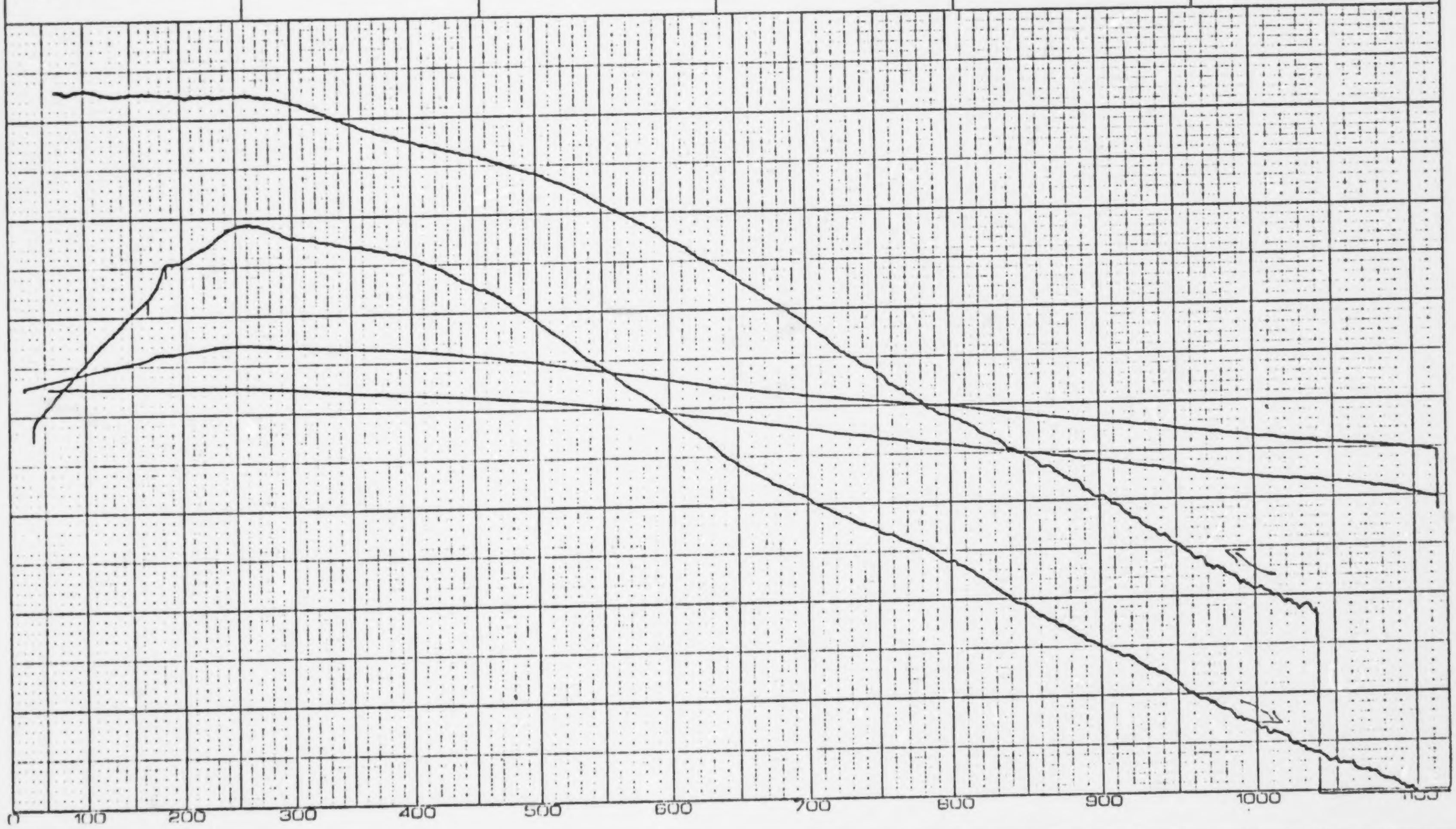
ATM 0.8% O2 in ___
FLOW RATE ___

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in 1 (+0.2)
WEIGHT, mg 31.25
REFERENCE
Aluminum Oxide

TGA
SCALE, mg/in ___
SUPPRESSION, mg ___
WEIGHT, mg ___
TIME CONST., sec ___
dY, (mg/min)/in ___

TMA
SCALE, mils/in ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___



DUPONT Instruments

PART NO. 990092

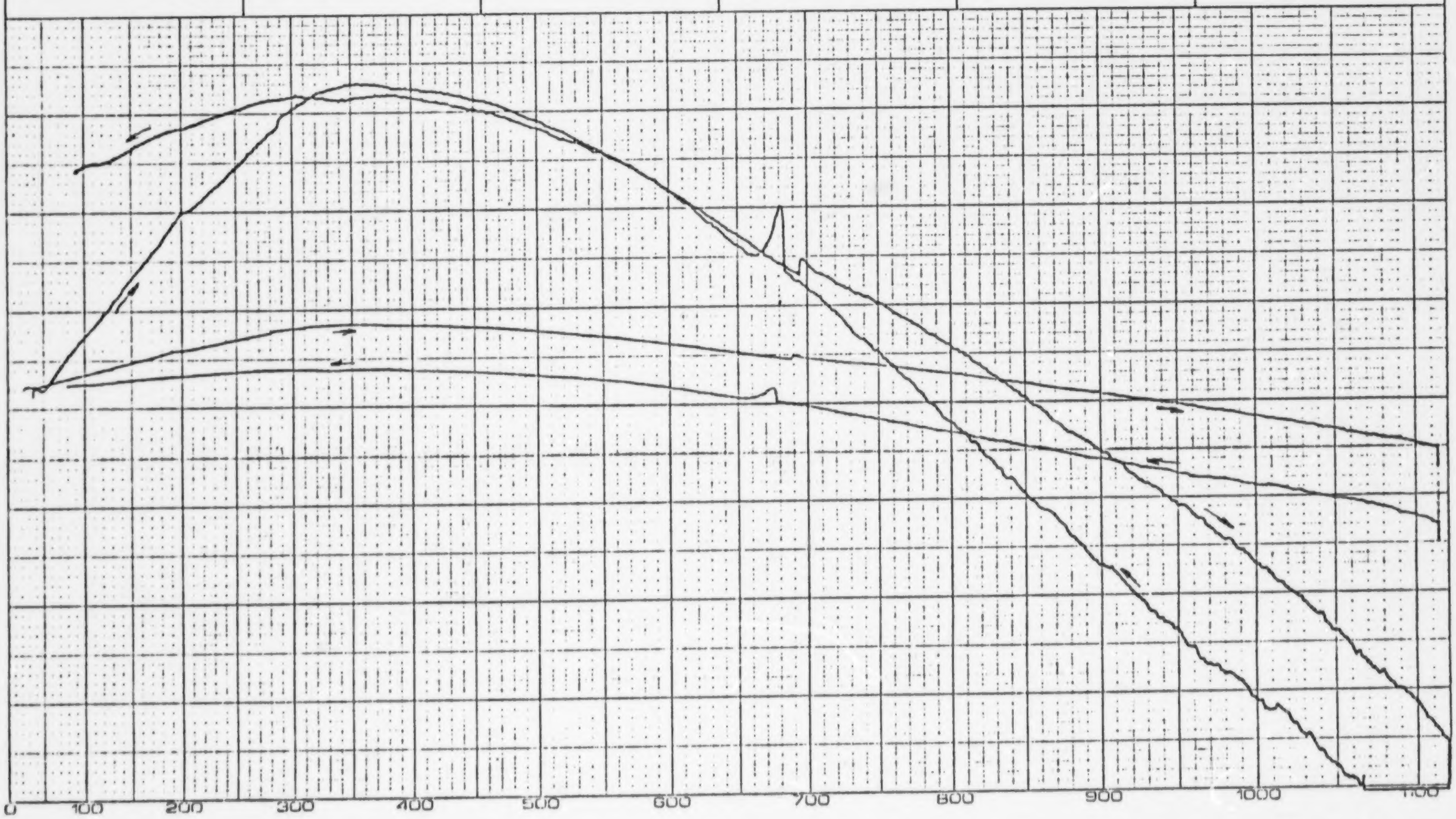
RUN NO.___ DATE 6/21/79
OPERATOR A. Pallay
SAMPLE: EPA 513
ATM 0.8% O₂
FLOW RATE ___

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT • COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in ___
WEIGHT, mg 32.90
REFERENCE
Aluminum Oxide

TGA
SCALE, mg/in ___
SUPPRESSION, mg ___
WEIGHT, mg ___
TIME CONST., sec ___
dY, (mg/min)/in ___

TMA
SCALE, mils/in ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___



MEASURED VARIABLE

DU PONT Instruments

PART NO. 990092

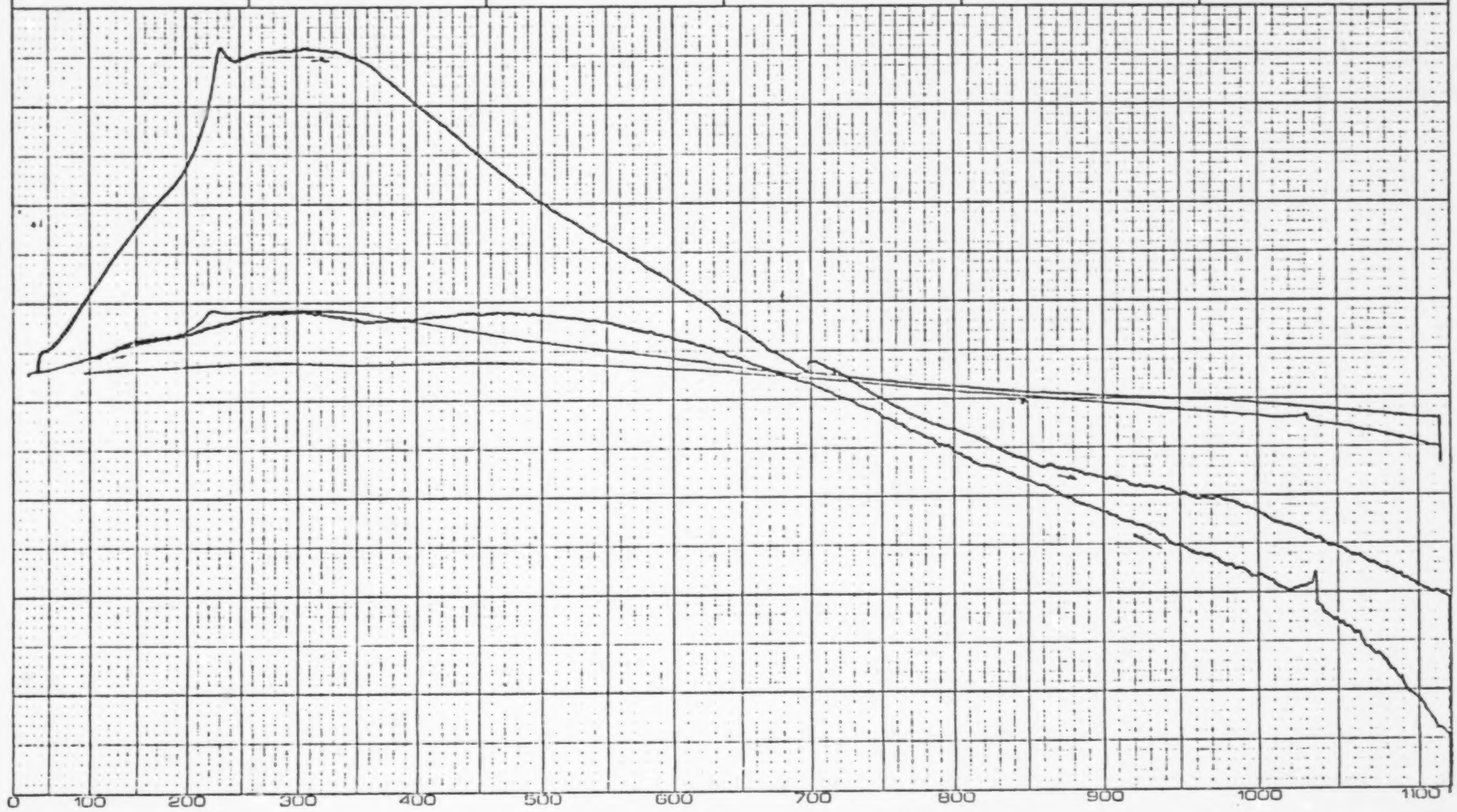
RUN NO.___ DATE 6/26/79
OPERATOR A. Pellat
SAMPLE:
CORNING - FORD
MONOLITH
ATMO: 8% O₂
FLOW RATE ___

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in ⊥ { 0.2
WEIGHT, mg 30.90
REFERENCE ___

TGA
SCALE, mg/in ___
SUPPRESSION, mg ___
WEIGHT, mg ___
TIME CONST., sec ___
dY, (mg/min)/in ___

TMA
SCALE, mils/in ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___



DU PONT Instruments

PART NO. 990092

RUN NO.___ DATE 6/27/79
OPERATOR A. Pillay
SAMPLE:
American Lava
Monolith.
ATM 0.3% O_2
FLOW RATE

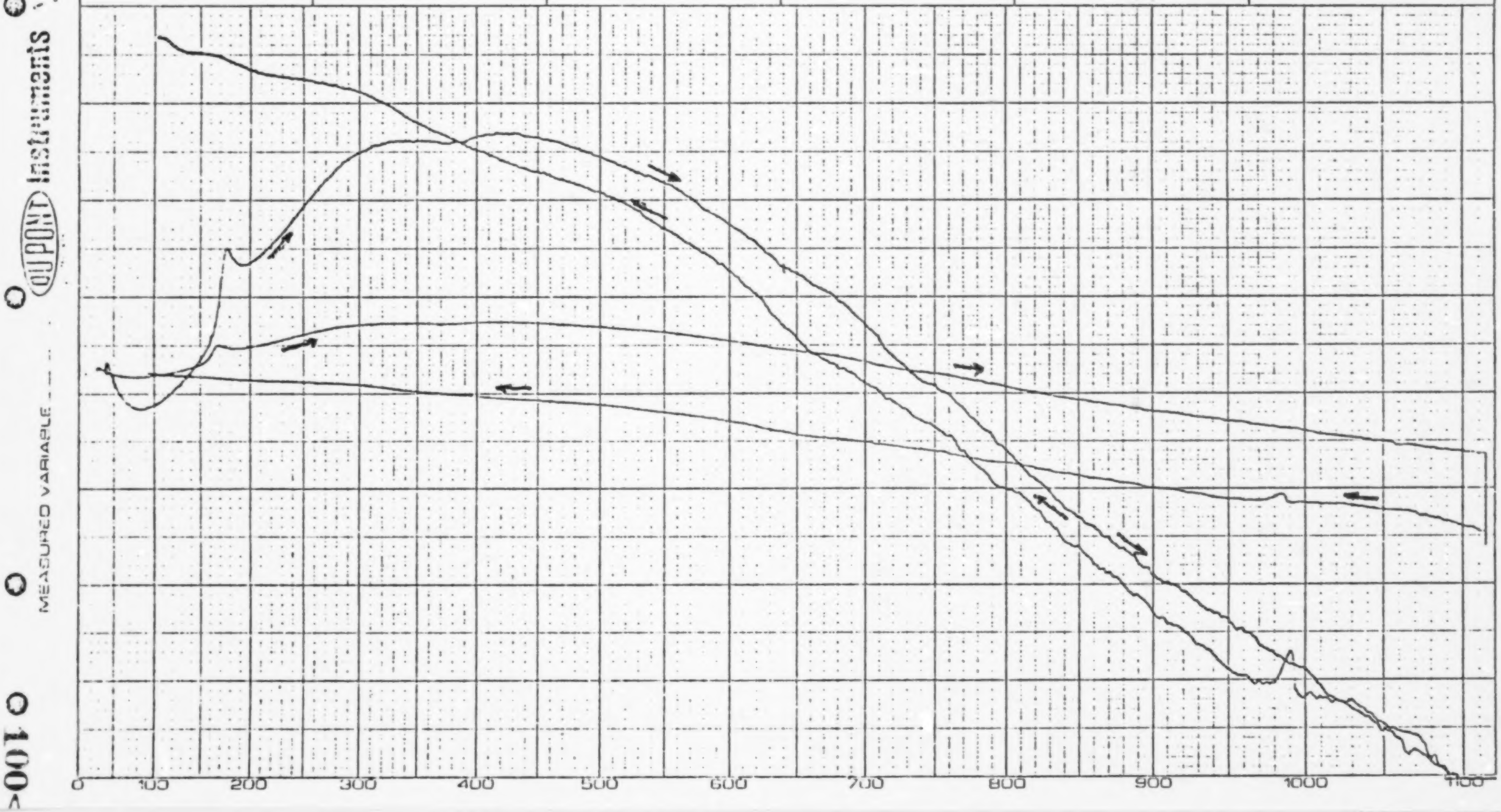
T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ISO
SHIFT, in 0

DTA-DSC
SCALE, °C/in
(mcal/sec)/in 1 & 0.2
WEIGHT, mg 32.08
REFERENCE
Aluminum Oxide

TGA
SCALE, mg/in
SUPPRESSION, mg
WEIGHT, mg
TIME CONST., sec.
dY, (mg/min)/in

TMA
SCALE, mils/in
MODE
SAMPLE SIZE
LOAD, g
dY, (10X), (mils/min)/in

DUPONT Instruments



PART NO. 990092

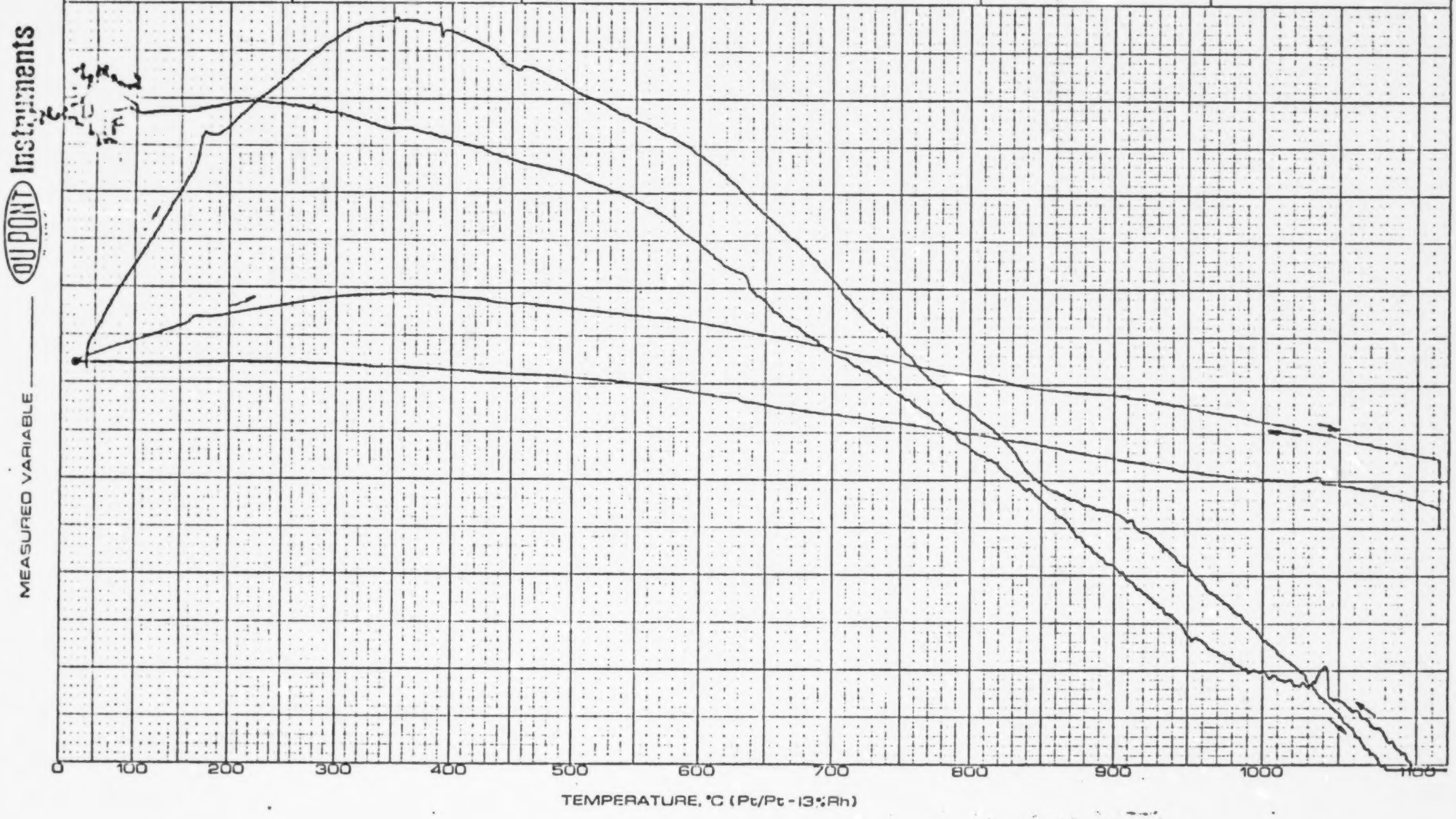
R NO. DATE 6/21/79
OPERATOR A. PALLAY
SAMPLE: EPA - 524
ATM 0.8% O_2
FLOW RATE

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ISO
SHIFT, in 0

DTA-DSC
SCALE, °C/in
(mcal/sec)/in 1 ↓0.2
WEIGHT, mg 32.03
REFERENCE
ALUMINUM OXIDE

TGA
SCALE, mg/in
SUPPRESSION, mg
WEIGHT, mg
TIME CONST., sec
dY, (mg/min)/in

TMA
SCALE, mils/in
MODE
SAMPLE SIZE
LOAD, g
dY, (10X), (mils/min)/in



DUPONT Instruments

PART NO. 990092

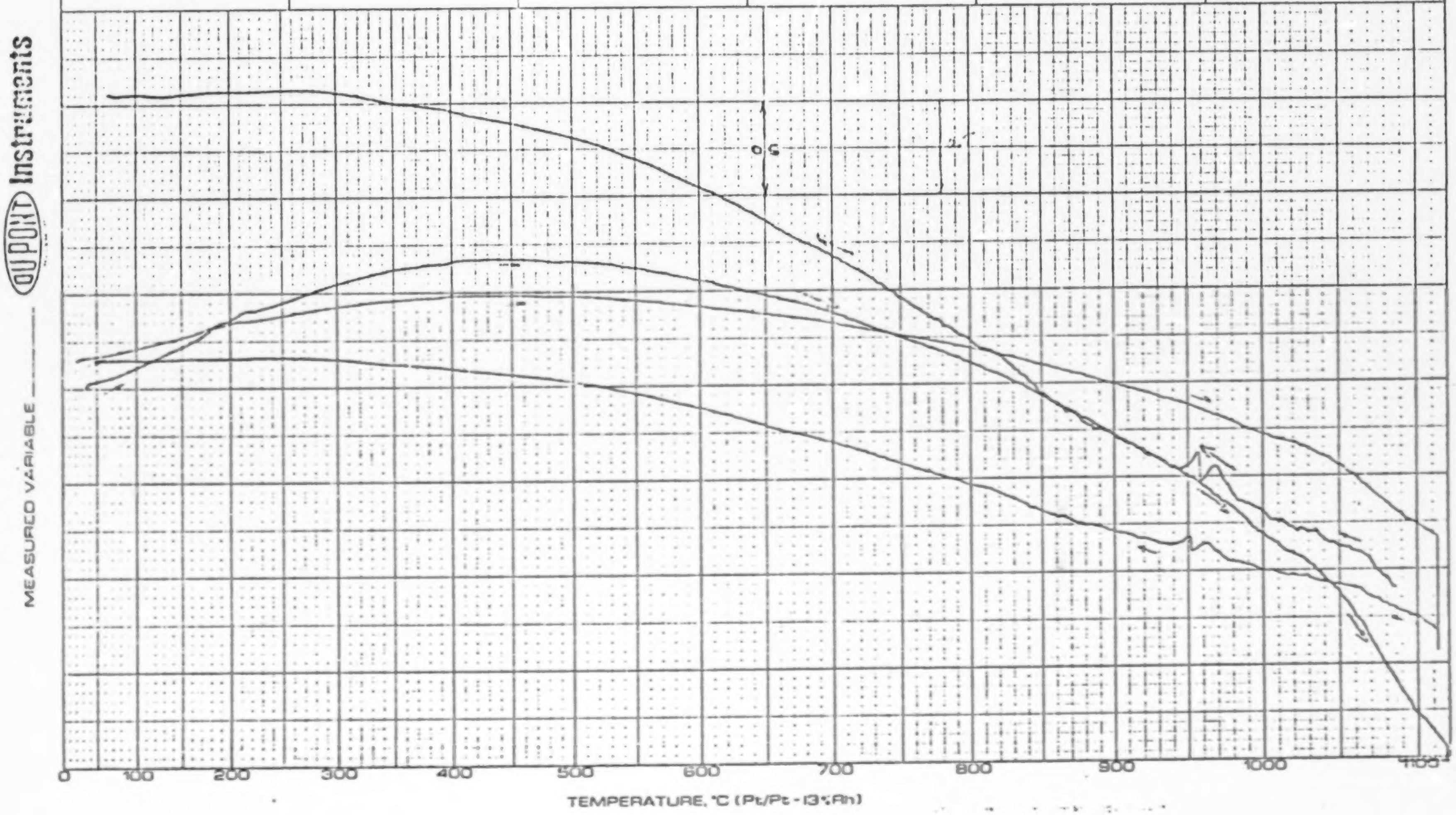
RUN NO. ___ DATE 6/29/79
OPERATOR A. Pilley
SAMPLE EPA 525 I
ATMO. 8% O₂ ...
FLOW RATE ___

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in Ė 0.5
WEIGHT, mg 33.26
REFERENCE
Aluminum Oxide

TGA
SCALE, mg/in ___
SUPPRESSION, mg ___
WEIGHT, mg ___
TIME CONST., sec ___
dY, (mg/min)/in ___

TMA
SCALE, mils/in ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___



PART NO. 990092

[illegible]

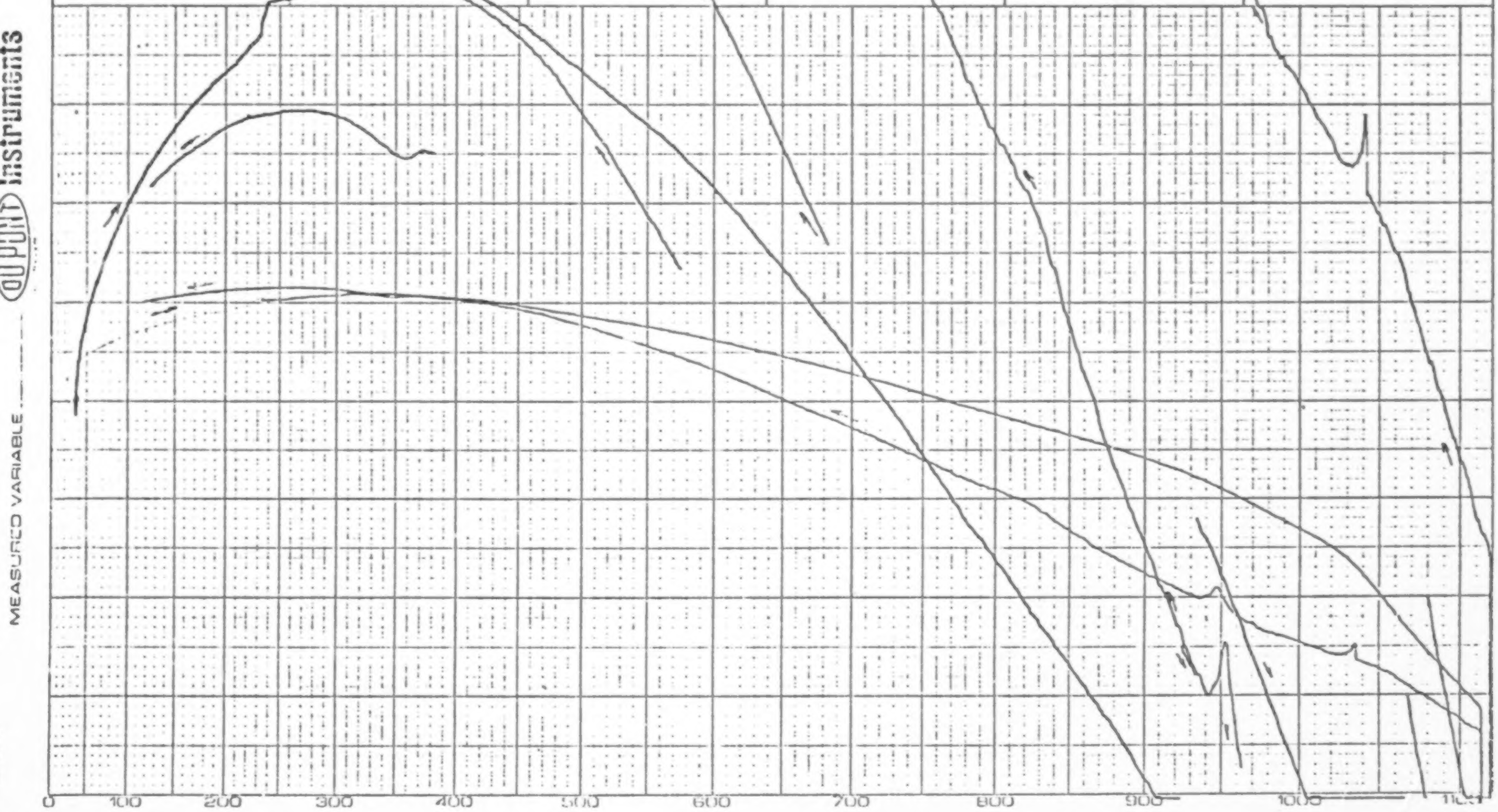
RUN NO. ___ DATE ______
OPERATOR A. Pillay
SAMPLE: EPA - 525
ATM 0.8% O_2 in ______
FLOW RATE ______

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ______
(mcal/sec)/in 1.902
WEIGHT, mg 32.63
REFERENCE ______
Aluminum Oxide

TGA
SCALE, mg/in ______
SUPPRESSION, mg ______
WEIGHT, mg ______
TIME CONST., sec ______
dY, (mg/min)/in ______

TMA
SCALE, mils/in ______
MODE ______
SAMPLE SIZE ______
LOAD, g ______
dY, (10X), (mils/min)/in ______

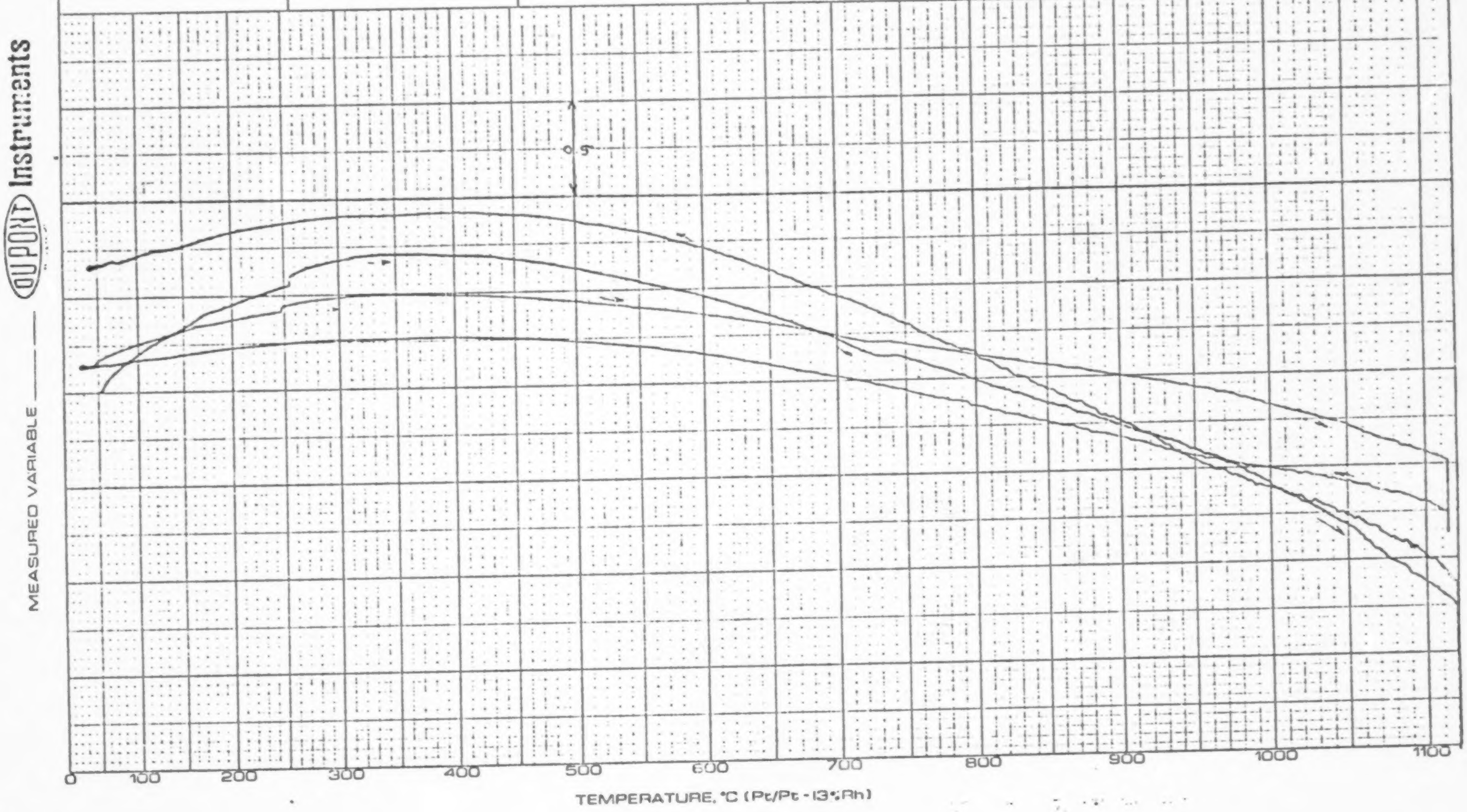


DUPONT Instruments

PART NO 990092

II

RU O.___ DATE 6/25/75	T-AXIS	DTA-DSC	TGA	TMA	
OPERATOR A. PALLAY	SCALE, mv/in 0.8	SCALE, °C/in ___	SCALE, mg/in ___	SCALE, mils/in ___	
SAMPLE EPA-526	PROG. RATE, °C/min 10	(mcal/sec)/in ↓0.5	SUPPRESSION, mg ___	MODE ___	
	HEAT ✓ COOL ___ ISO ___	WEIGHT, mg 51.75	WEIGHT, mg ___	SAMPLE SIZE ___	
ATM 0.8% O₂ ___	SHIFT, in 0	REFERENCE ___	TIME CONST., sec ___	LOAD, g ___	
FLOW RATE ___	___	ALUMINUM OXIDE	dY, (mg/min)/in ___	dY, (10X), (mils/min)/in ___	



DU PONT Instruments

PART NO. 990092

RUN NO. ___ DATE 6/22/79
OPERATOR A. PILLAY
SAMPLE: EPA - 526

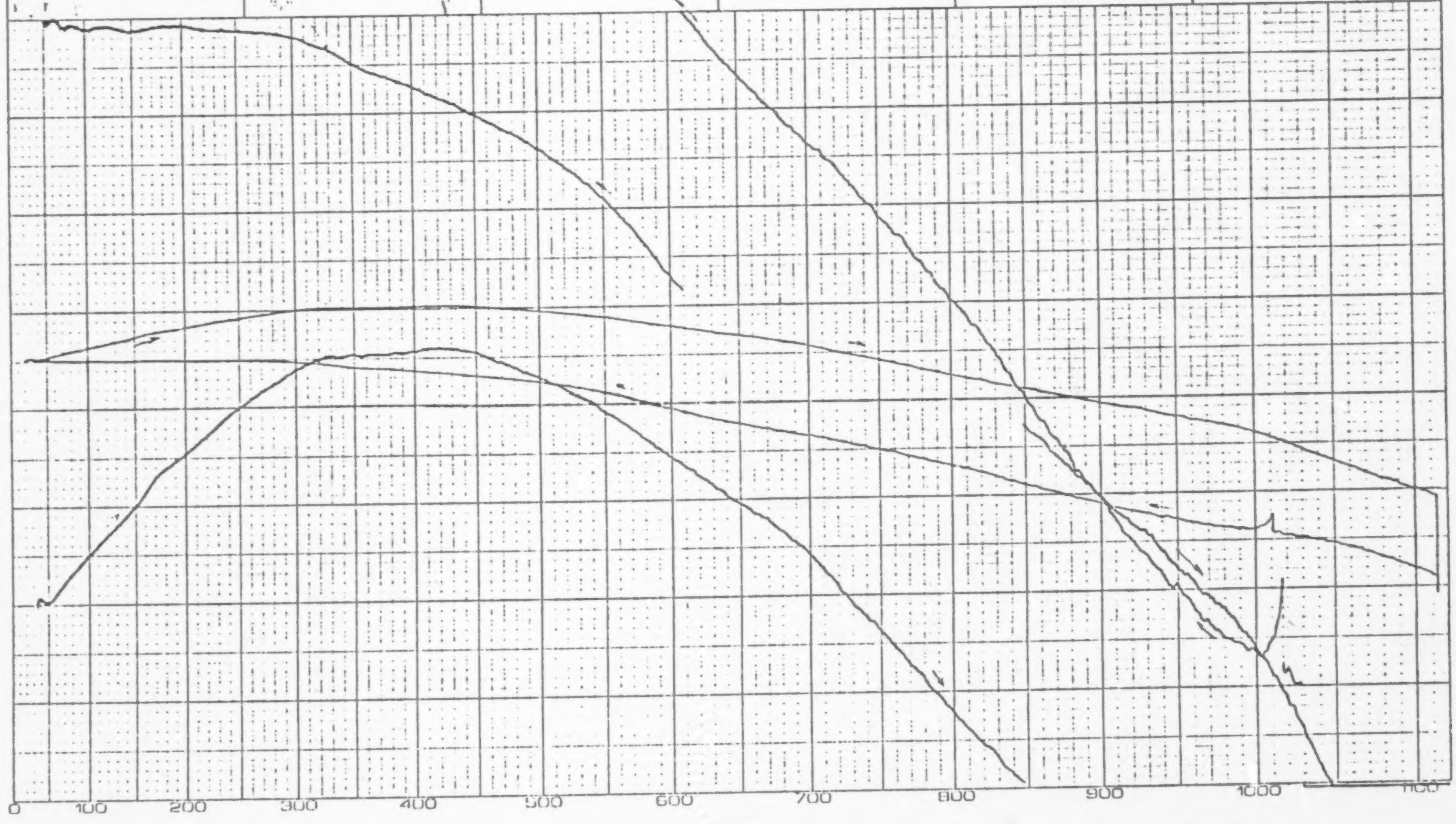
ATM 0.8% O₂ (in ___
FLOW RATE ___

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in 1 & 0.2
WEIGHT, mg 32.66
REFERENCE ___
ALUMINUM OXIDE

TGA
SCALE, mg/in ___
SUPPRESSION, mg ___
WEIGHT, mg ___
TIME CONST., sec ___
dY, (mg/min)/in ___

TMA
SCALE, mils/in ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___



MEASURED VARIABLE

DU PONT Instruments

PART NO. 990092

RUN NO.__ DATE 6/26/79
OPERATOR A. Pallay
SAMPLE: Ethyl - Ac Pellets
Control Samp
ATM 0.8% Oil
FLOW RATE

T-AXIS

SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL __ ISO __
SHIFT, in 0

DTA-DSC

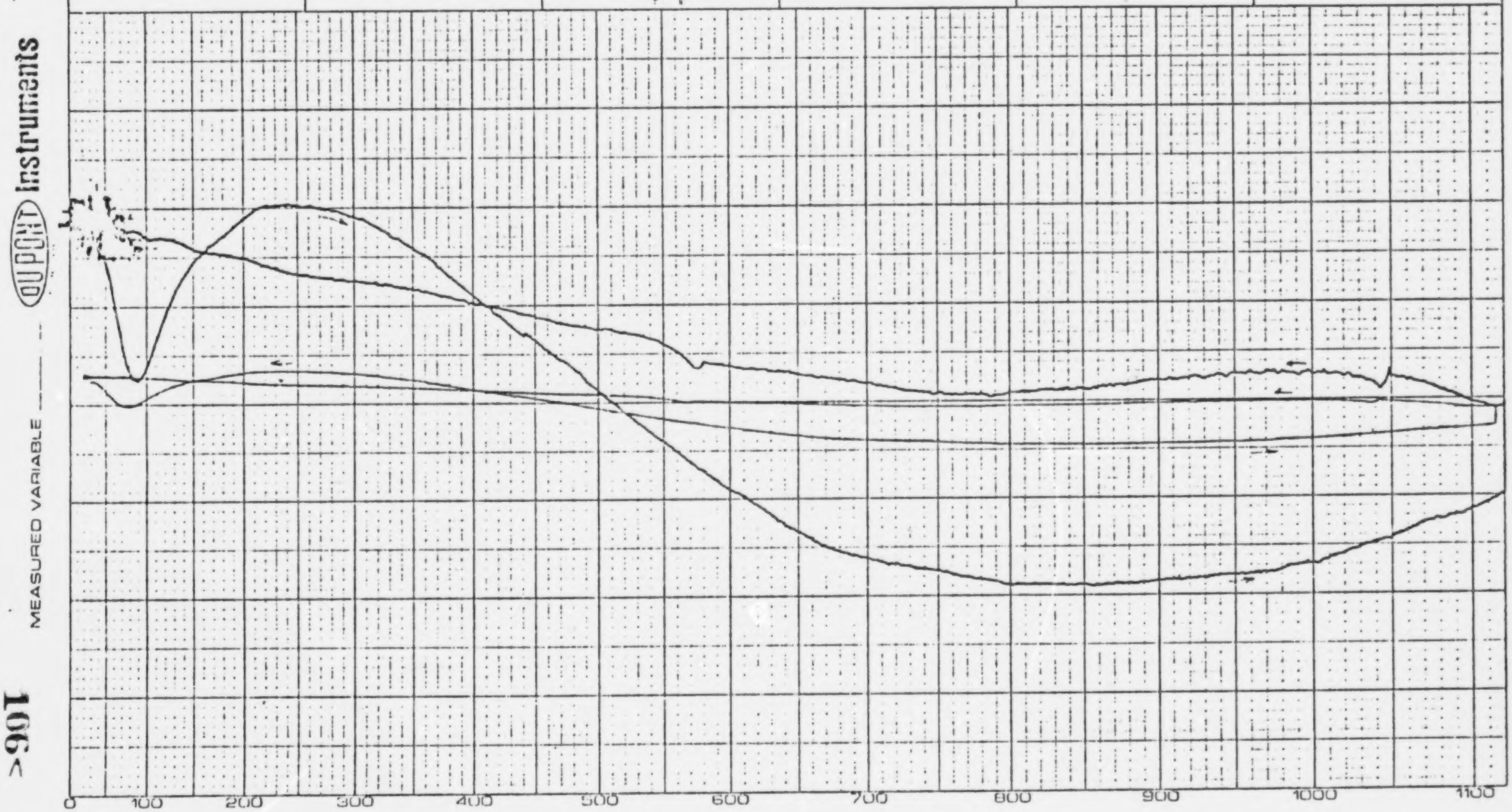
SCALE, °C/in
(mcal/sec)/in 1 & 0.2
WEIGHT, mg 29.20
REFERENCE
Aluminum Oxide

TGA

SCALE, mg/in
SUPPRESSION, mg
WEIGHT, mg
TIME CONST., sec
dY, (mg/min)/in

TMA

SCALE, mils/in
MODE
SAMPLE SIZE
LOAD, g
dY, (10X), (mils/min)/in

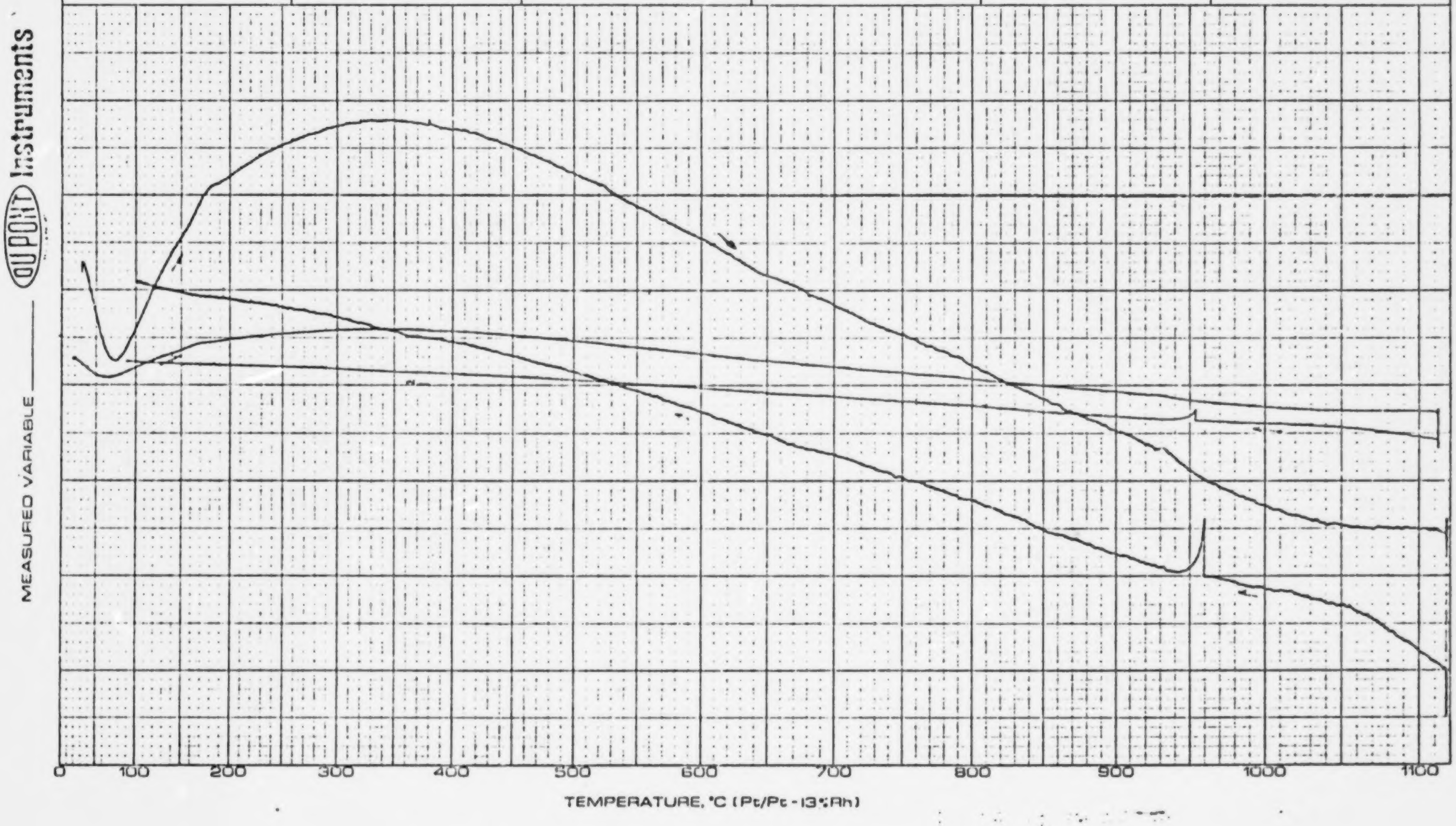


DUPONT Instruments

PART NO. 990092

RUN NO. ___ DATE ___	**T-AXIS**	**DTA-DSC**	**TGA**	**TMA**	
OPERATOR A Rucay	SCALE, mv/in 0.8	SCALE, °C/in ___	SCALE, mg/in ___	SCALE, mils/in ___	
SAMPLE: EPA - 537 [illegible]	PROG. RATE, °C/min 10	(mcal/sec)/in ± 0.2	SUPPRESSION, mg ___	MODE ___	
	HEAT ✓ COOL ___ ISO ___	WEIGHT, mg 29.64	WEIGHT, mg ___	SAMPLE SIZE ___	
ATM 0.8% [illegible]	SHIFT, in 0	REFERENCE	TIME CONST., sec ___	LOAD, g ___	
FLOW RATE ___		Aluminum Oxide	dY, (mg/min)/in ___	dY, (10X), (mils/min)/in ___	

DU PONT Instruments



PART NO. 990092

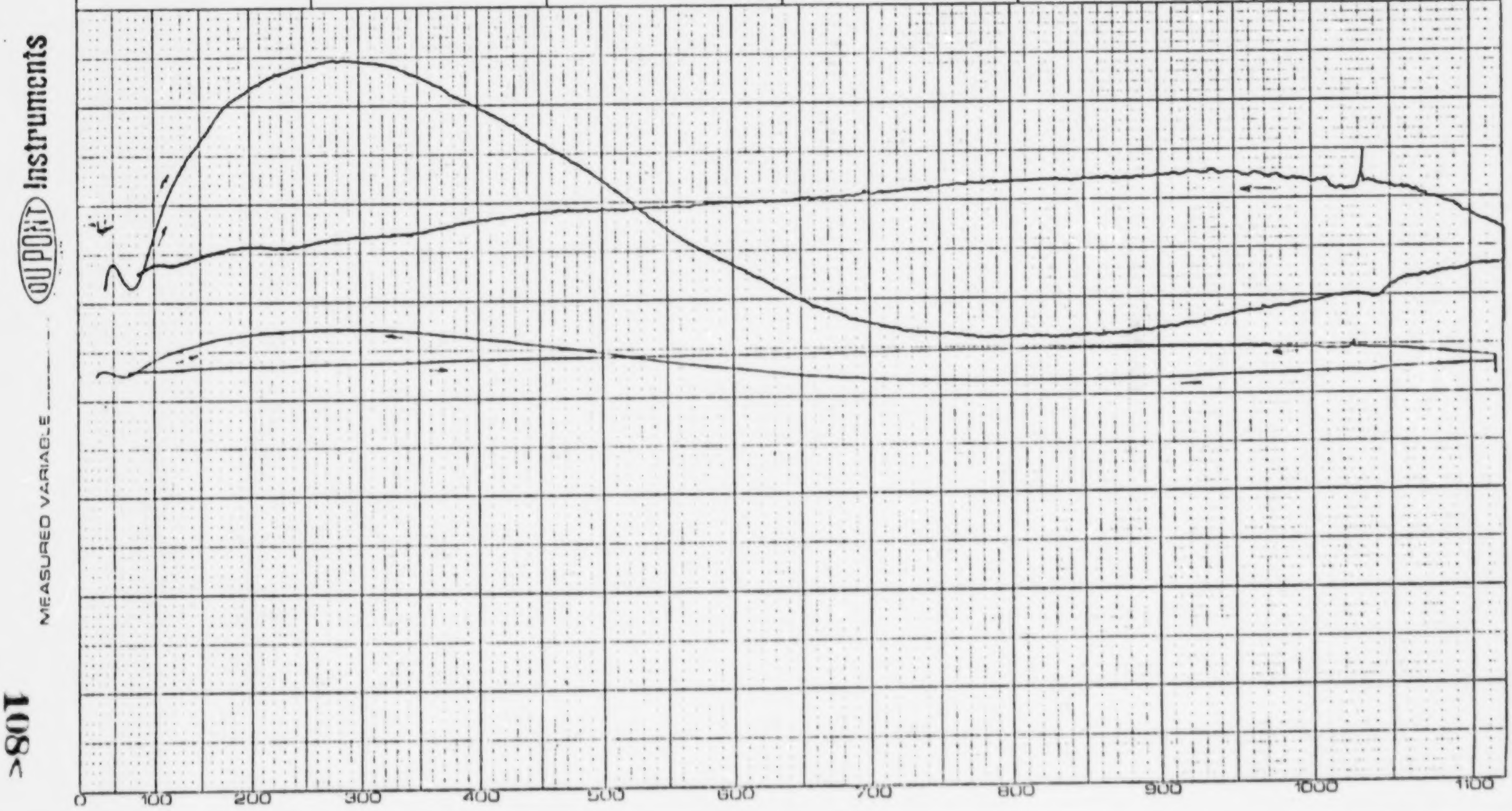
RUN NO.__ DATE 8/25/79
OPERATOR A. Rulay
SAMPLE: EPA - 538
Sylvanol
ATM 0.8% O_2 in [illegible]
FLOW RATE

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ISO
SHIFT, in 0

DTA-DSC
SCALE, °C/in
(mcal/sec)/in 1 & 0.2
WEIGHT, mg 27.26
REFERENCE
Aluminum Oxide

TGA
SCALE, mg/in
SUPPRESSION, mg
WEIGHT, mg
TIME CONST., sec
dY, (mg/min)/in

TMA
SCALE, mils/in
MODE
SAMPLE SIZE
LOAD, g
dY, (10X), (mils/min)/in



DUPONT Instruments

PART NO. 970092

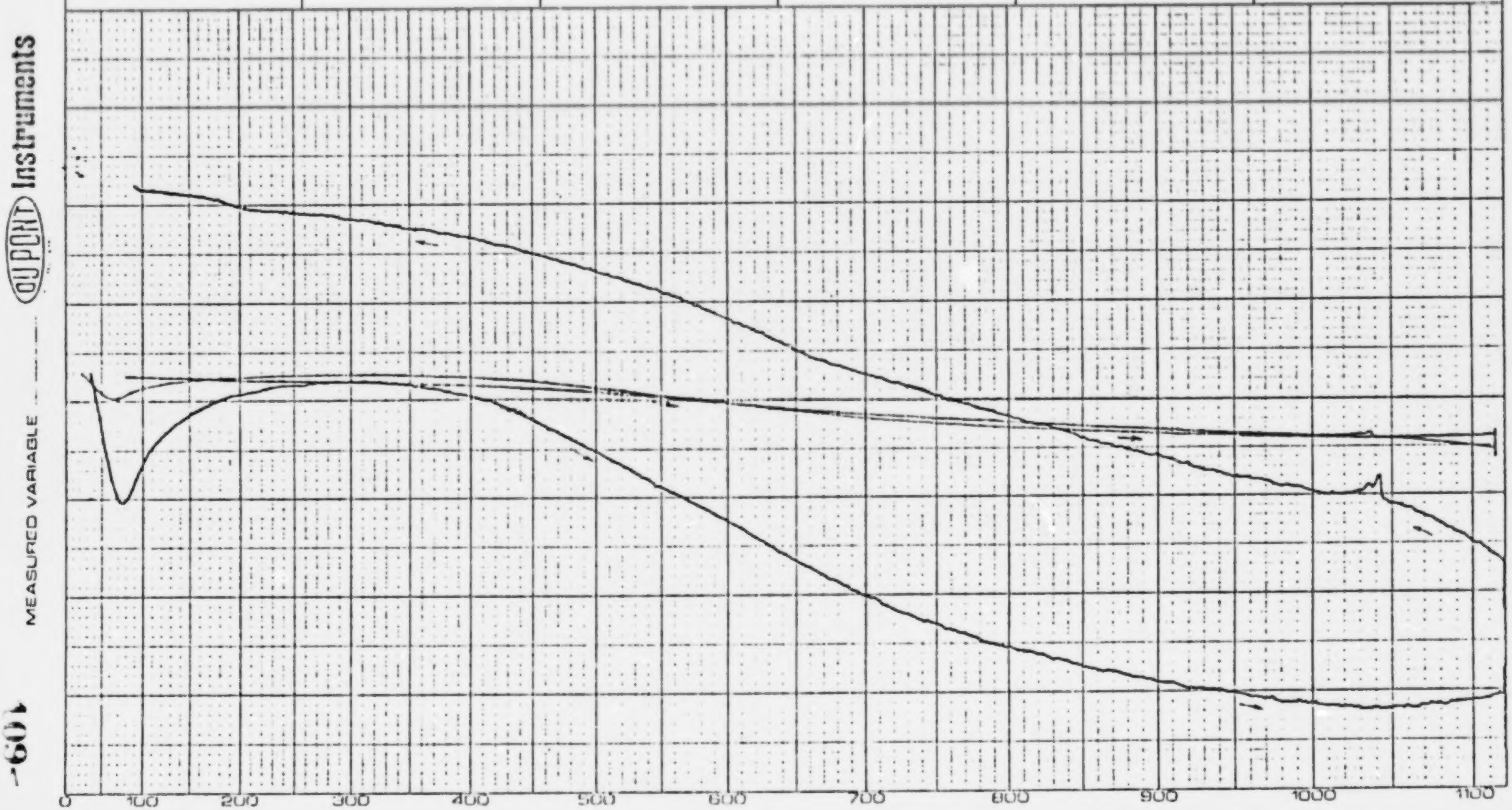
RUN NO.___ DATE 6/26/79
OPERATOR A. Pallay
SAMPLE: EPA 539
Sylvania
ATM 0.5% O₂ in ______
FLOW RATE ______

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 10
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ______
(mcal/sec)/in 1 / 0.2
WEIGHT, mg 31.72
REFERENCE ______

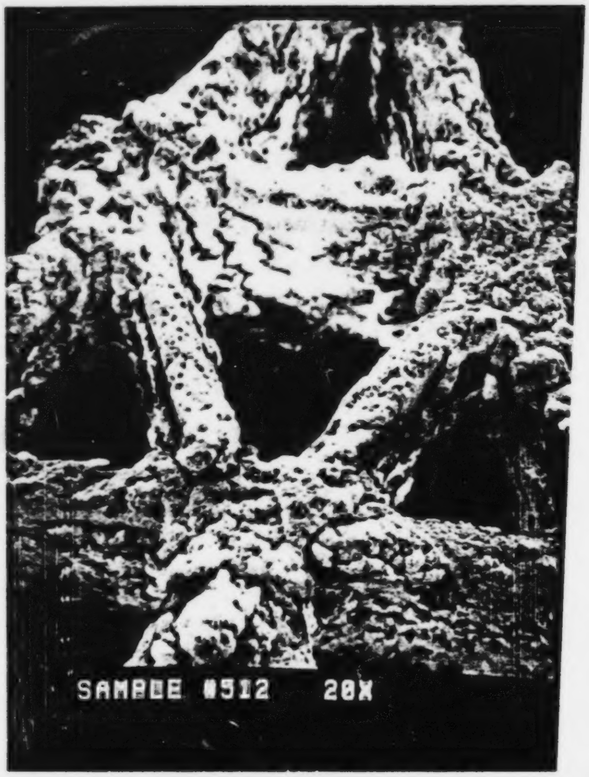
TGA
SCALE, mg/in ______
SUPPRESSION, mg ______
WEIGHT, mg ______
TIME CONST., sec ______
dY, (mg/min)/in ______

TMA
SCALE, mils/in ______
MODE ______
SAMPLE SIZE ______
LOAD, g ______
dY, (10X), (mils/min)/in ______



DU PONT Instruments

H. SCANNING ELECTRON MICROGRAPHS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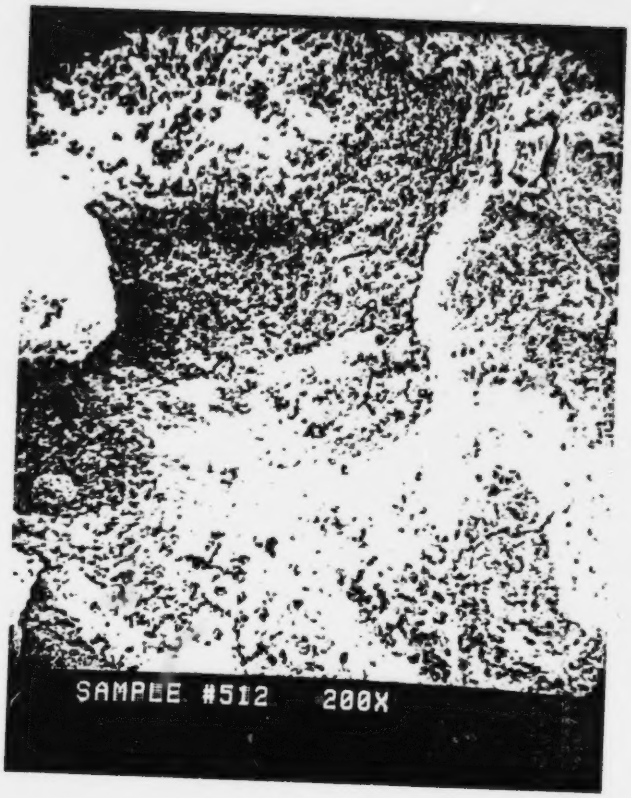
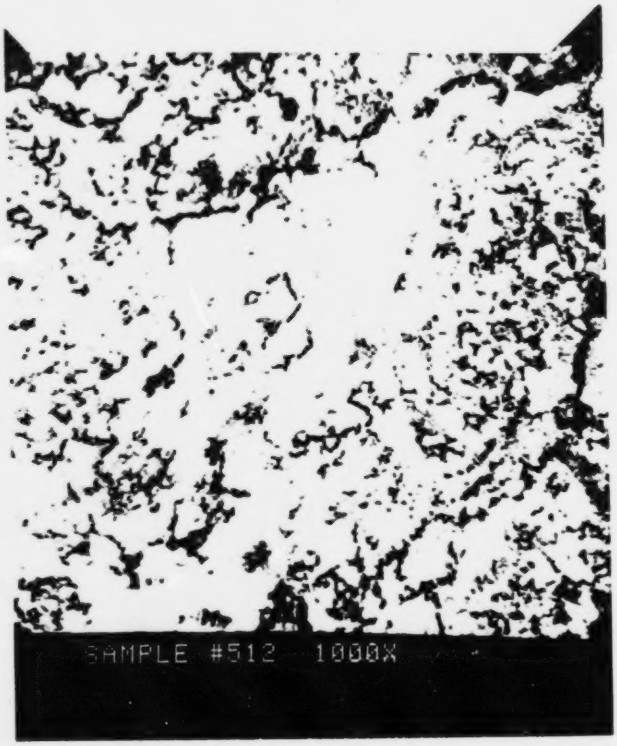

SAMPLE #512 200X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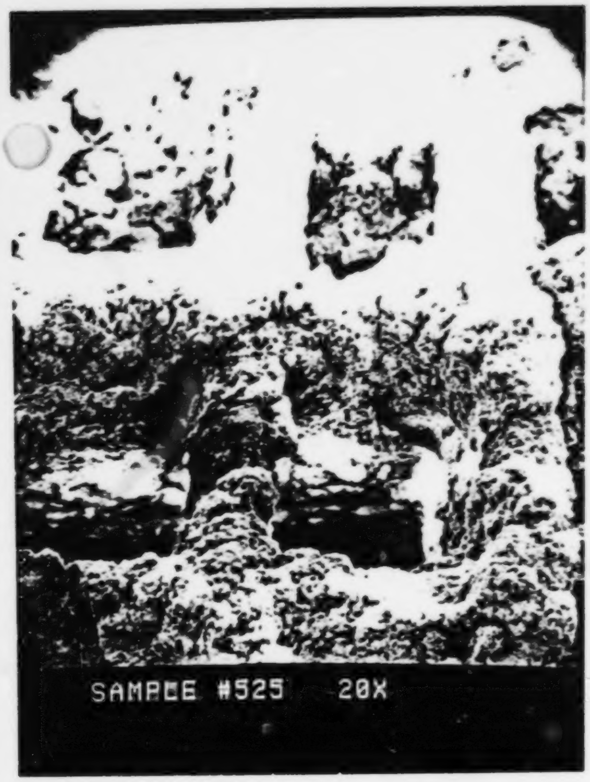
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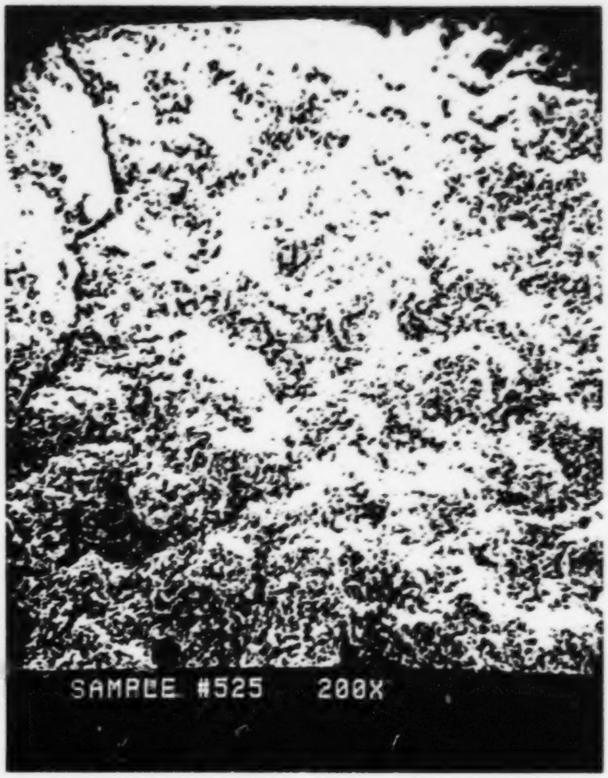
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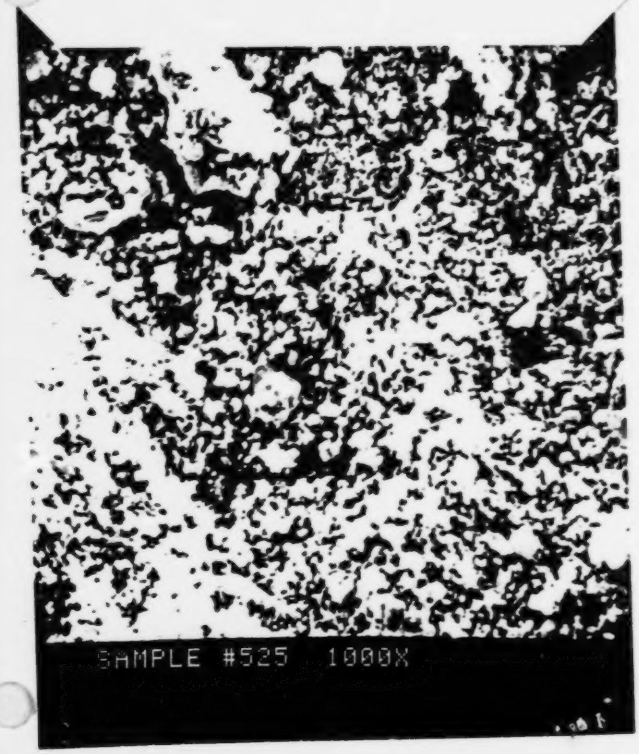
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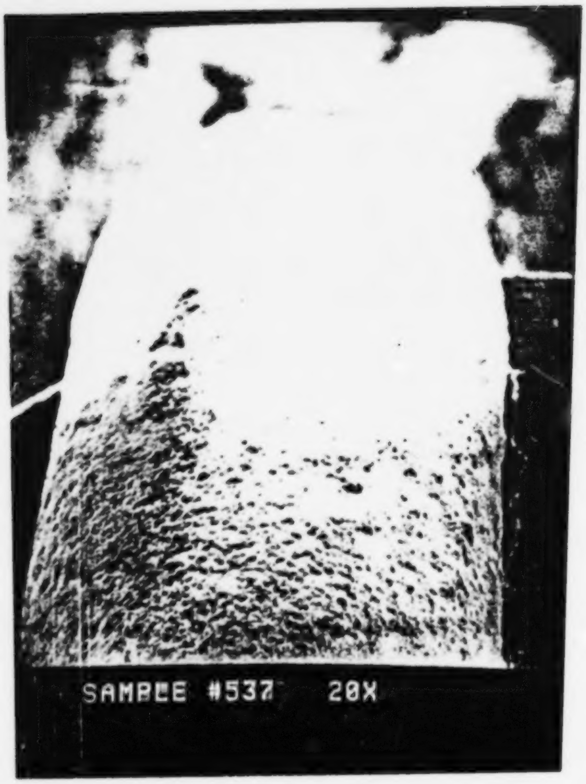
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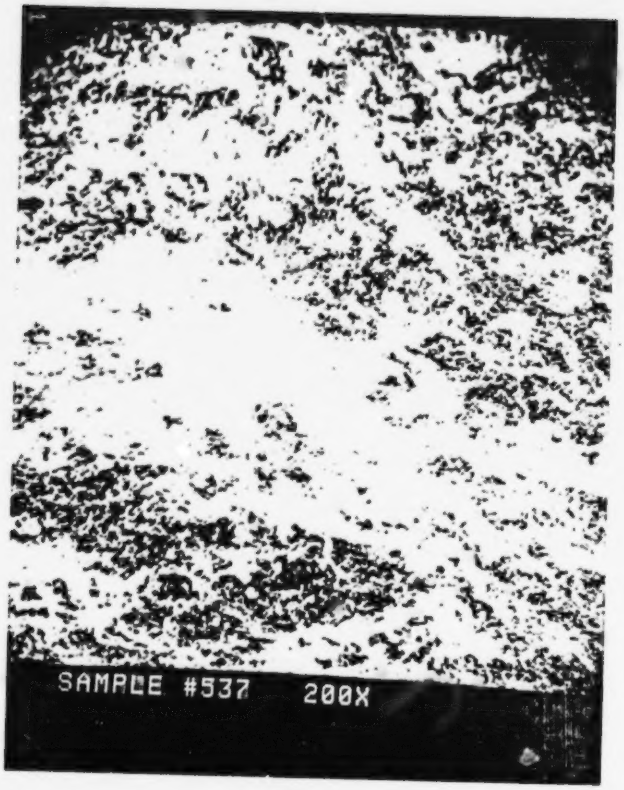
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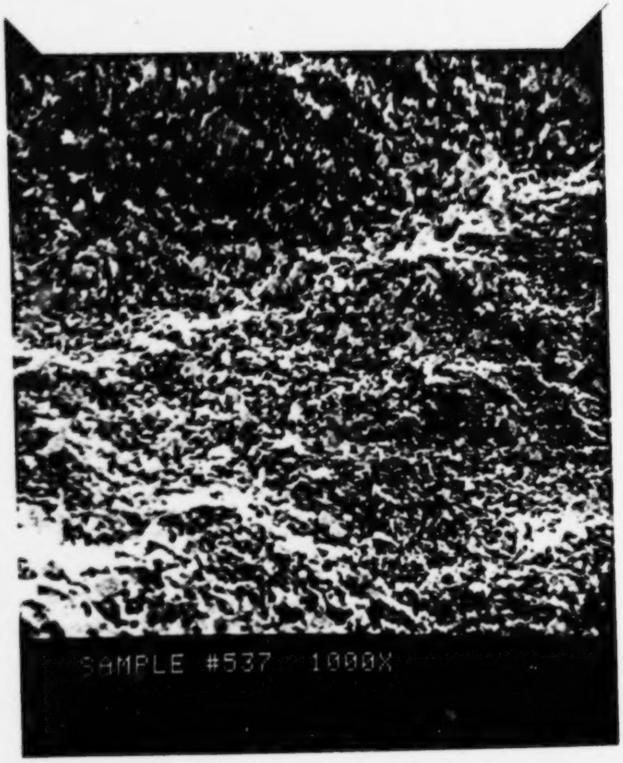
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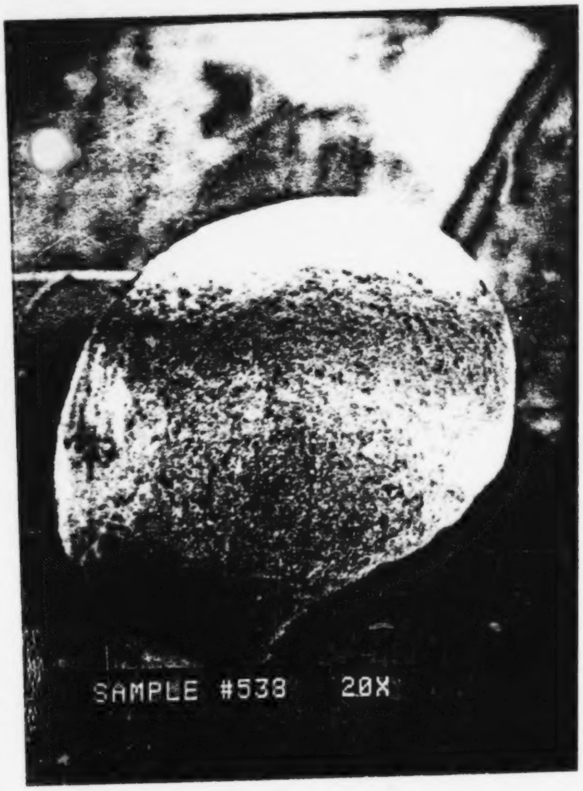
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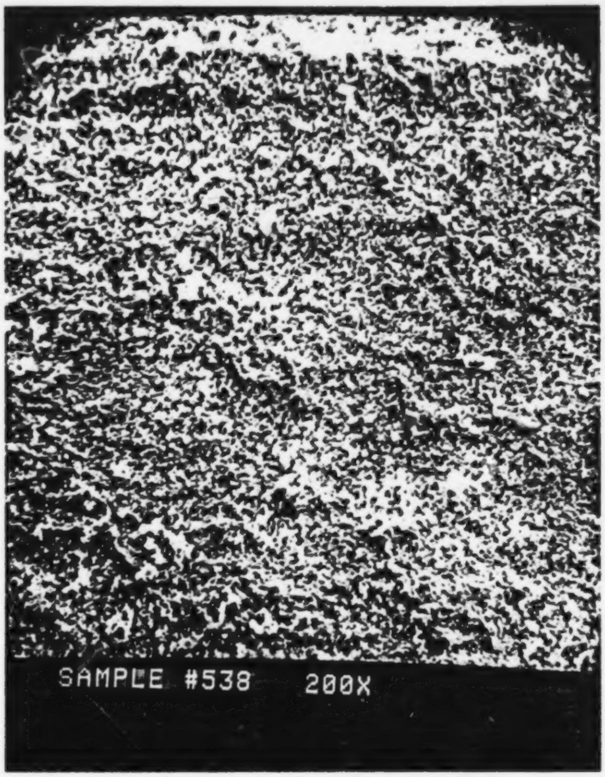
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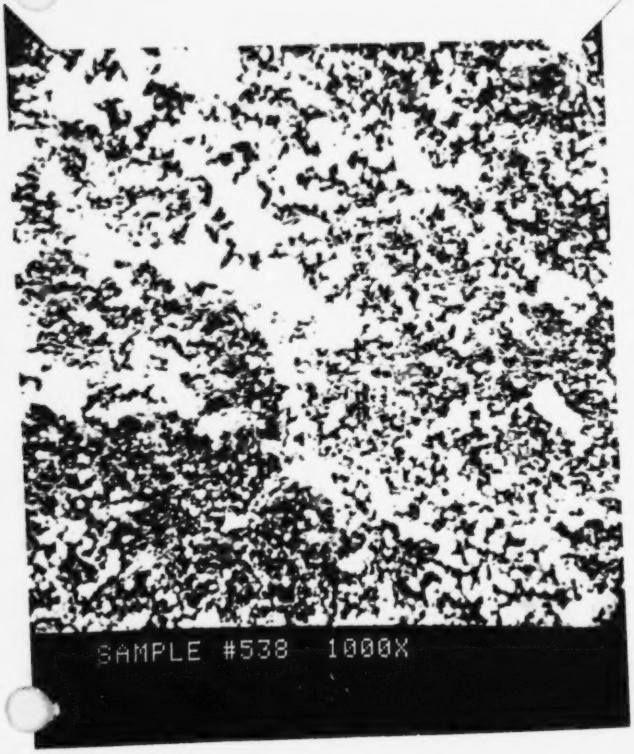
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